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**DESIGN, FABRICATION AND  
TESTING OF A WET OXIDATION  
WASTE PROCESSING SYSTEM**

**MAY 15, 1975**

**Prepared Under Contract NAS 1-11748**

**by**

**BIOENGINEERING ORGANIZATION  
LOCKHEED MISSILES & SPACE COMPANY  
SUNNYVALE, CALIFORNIA**

**for**

**NATIONAL AERONAUTICS & SPACE ADMINISTRATION  
JOHNSON SPACE CENTER**

(NASA-CF-141916) DESIGN, FABRICATION AND  
TESTING OF A WET OXIDATION WASTE PROCESSING  
SYSTEM Final Report (Lockheed Missiles and  
Space Co.) 116 p HC \$5.25 CSCI 06K

**N75-28721**

**Unclas**

**31071**

**G3/54**



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## DESIGN, FABRICATION AND TESTING OF A WET OXIDATION WASTE PROCESSING SYSTEM

### SUMMARY

Wet oxidation has been used commercially for sewage sludge oxidation for several decades. NASA interest in the process for spacecraft application results from the fact that waste can be oxidized in the presence of oxygen, water, and steam at temperatures of  $505^{\circ}\text{K}$  ( $450^{\circ}\text{F}$ ) to  $560^{\circ}\text{K}$  ( $550^{\circ}\text{F}$ ) and pressures from  $6.9 \text{ MN/m}^2$  (1000 psi) to  $15.2 \text{ MN/m}^2$  (2200 psi) without producing the gaseous oxides of nitrogen, sulphur, and phosphorous, while producing a clear, salty, sterile effluent water and white phosphate ash. This allows recovery of useful water and gases from a wide variety of spacecraft wastes and the elimination of all overboard venting.

Previous development efforts: 1) explored process conditions for spacecraft application; 2) constructed a breadboard reactor system and demonstrated the feasibility of operating the system continuously; 3) began the development of component designs; and 4) investigated potential improvements in the process.

The contract work covered by this report was based on the previous developments and provided for the detail design, fabrication and testing of an engineering prototype wet oxidation system. The testing was intended to demonstrate the component and system performance in a 45 day test. The system functional design that evolved from the previous development efforts consisted of the following components or subsystems:

- o A trash pulverizer subsystem to produce a pumpable slurry from miscellaneous spacecraft solid wastes.
- o A slurry pumping subsystem to provide for accumulation of pulverized trash and feces and urine; the pressurization of the slurry to operating pressure, and its introduction into the reactor system to maintain a continuous flow.

- o An oxygen supply subsystem consisting of high pressure storage, pressure regulation, and flow control.
- o A catalyst introduction subsystem to deliver a small, measured flow of catalyst into the slurry feed to prevent ammonia formation and promote the oxidation of wastes in the reactor.
- o A regenerative heat exchanger to heat the incoming oxygen, slurry with the hot liquid and gas effluent from the reactor as a heat source.
- o A heated, stirred reactor to provide for oxidation of the waste materials.
- o Pressure controls to maintain the reactor at operating pressure.
- o Filters to remove the finely divided ash produced by the oxidation process.
- o A phase separator to separate the product liquid and gas.
- o Controls and instrumentation to provide automatic operation and fail-safe shutdown.

The system described above was fabricated and assembled into two modules: the slurry and oxygen supply module; and the process and water recovery module. The two modules were set side by side, connected to each other and to supporting laboratory equipment. Following checkout and calibration tests, a 45 day test was run based on continuous operation from Monday morning through Friday afternoon. 737 hours of testing were completed with the following results.

- o The system produced effluent water which was clear, pale yellow with a Total Organic Carbon (TOC) value of from 200 to 300 mg/liter. The color resulted from the catalyst. The water quality was such that passing it through a vapor compression distillation unit and charcoal should provide potable water.
- o The trash pulverizer was incapable of providing a pumpable trash slurry, so that all but 23 hours of testing was run with only fecal/urine slurry.
- o The slurry pump, oxygen supply, catalyst introduction, heat exchanger, pressure regulator, filter, phase separator, and control systems worked well throughout the test program with a few minor difficulties.

- o The reactor bearings located directly in the heated slurry test program.
- o The catalyst was originally expected to pass through the reactor and the system. However, it deposited instead on the reactor surface and acted as a permanent catalyst. Catalyst introduction was stopped after 78 hours of testing and no change in effluent characteristics was noted during the remaining 659 hours of testing.
- o The reactor inlet porting caused a flow blockage four times during the test.
- o Pulverized dry waste was introduced into the system during the last 23 hours of testing and clogged the supply line in several places and the reactor inlet port.

General conclusions based on these test results are:

- o Redesign of the reactor is needed to provide increased bearing life and an unobstructed slurry input path.
- o Catalyst introduction can be limited to an initial reactor "pickling period".
- o A major design and development effort is required to develop a dry waste pulverizer subsystem capable of producing a pumpable slurry. Some changes in the waste model may be required to allow automatically controlled dry waste pulverization with reasonable power.
- o Dry waste slurry of a solids concentration consistent with the load model will require: a minimum of 0.95 cm (3/8") tubing and fittings in cool areas of the process; no sharp or straight edges across tubing flow paths; straight through flow path in all fittings; ball valves in the flow path; smooth and gradual neck-down design for inlets and outlets for tankage; and piston rather than bellows type design for variable volume tankage.

## INTRODUCTION

The wet oxidation process has been developed for commercial use in the chemical, paper, and sewage treatment industries. It provides a low temperature, sometimes self-sustaining means of oxidizing wastes to relatively safe inert effluent liquid and gaseous products. Figure 1 is a schematic of a typical commercial wet oxidation process. The waste liquid is pumped to reactor pressure by a positive displacement slurry pump. Air from an air compressor is mixed with the slurry at the pump outlet, and the mixture passes through two regenerative heat exchangers before entering the reactor. Reactor conditions are generally in the range of  $505^{\circ}\text{K}$  ( $450^{\circ}\text{F}$ ) to  $560^{\circ}\text{K}$  ( $550^{\circ}\text{F}$ ) with corresponding pressures from  $6.9 \text{ MN/m}^2$  (1000 psi) to  $20.7 \text{ MN/m}^2$  (3000 psi). The effluent passes through the high temperature regenerative heat exchanger and to a liquid/vapor phase separator. The liquid is passed through the low temperature regenerative heat exchanger to heat the incoming slurry air mixture before discharge. The vapor and gases are fed to a mixed gas turbine that drives the feed air compressor allowing recovery of process energy. The primary advantage of the process for commercial applications is the low oxidation temperature that allows more economical burning of wastes and longer equipment operating life while producing a sterile, nearly inert liquid phase and a relatively clean gaseous phase.

Although the commercial units designed for low installation and operation costs are never operated to obtain extremely high degrees of oxidation, the process is capable of achieving 99 percent or greater reduction in chemical oxygen demand (COD) without producing the oxides of sulfur, phosphorous, and nitrogen that are associated with the more conventional high-temperature dry incineration. These elements appear as sulfate and phosphate ions in the liquid phase and can, therefore, be more readily handled. The purity of the liquid and gas phases are the primary reasons for interest in the process for manned spacecraft. The recovery of useful water and gases from urine and fecal matter



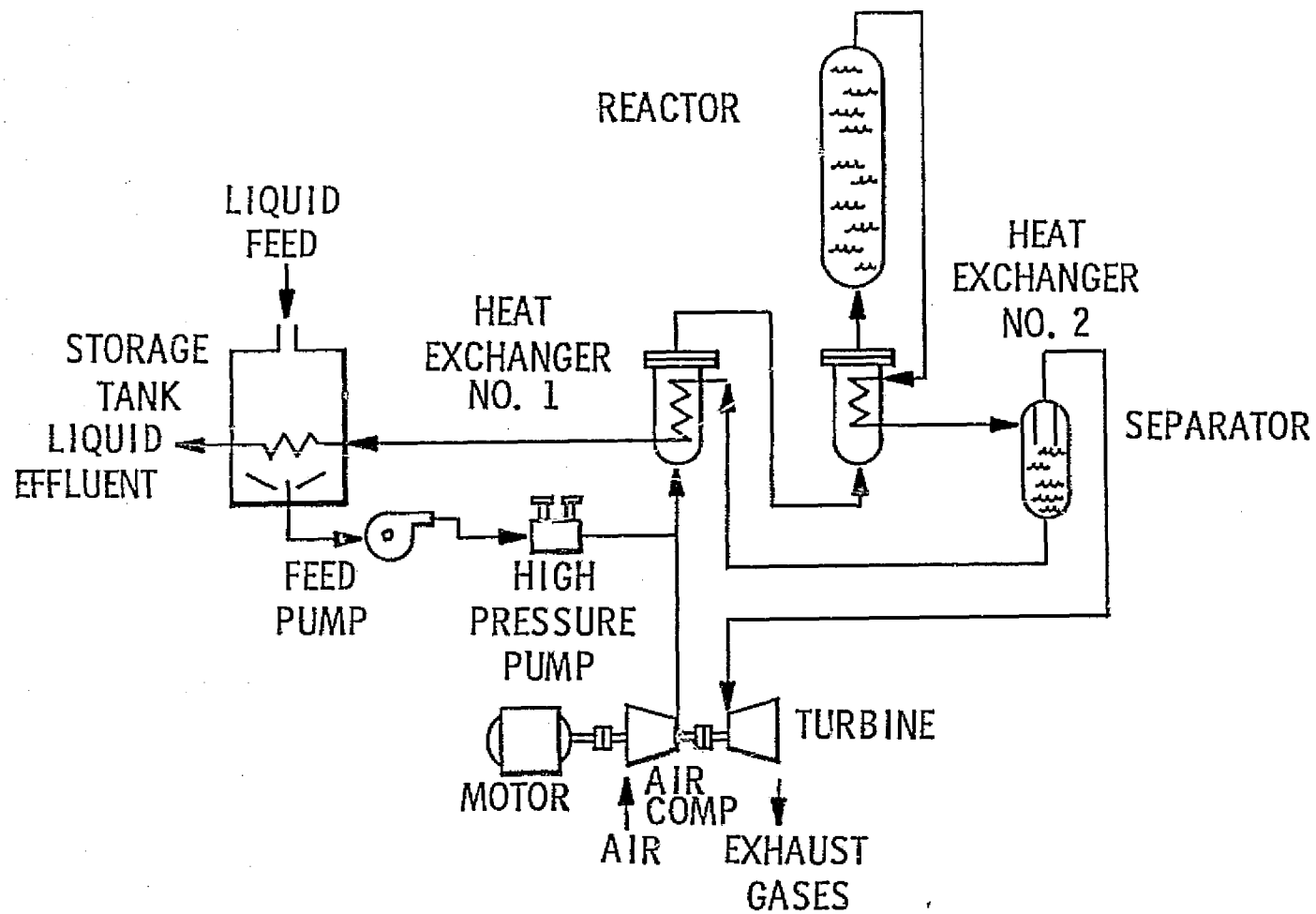


Fig. 1 Typical Commercial Wet Oxidation System

coincident with processing of wastes to a low volume sterile, nondegradable ash, and the elimination of overboard venting of waste liquids and gases are the promises of wet oxidation. The penalties that must be paid are the weight and power required for a moderate temperature, high pressure process, and the equipment problems of handling sewage sludge and salt water effluent.

The spacecraft wet oxidation process varies from the commercial process in a number of significant ways. The process flows are minute, pure oxygen is the preferred input gas, and the process must be optimized to yield the highest practical water and gas purity. It was recognized by NASA that a laboratory program was needed to investigate the feasibility of wet oxidation for spacecraft waste treatment.

Contract NAS 1-5295 was let to this end, and the results are reported (Ref. 1). The most significant conclusions are summarized in the following:

1. 95 percent or greater COD reduction was achieved.
2. Pure oxygen was preferable to air.
3. A base metal oxide catalyst showed promise for reducing temperature and promoting more complete oxidation at the higher temperature.
4. The effluent consisted of a dark, rapidly settling precipitate and a clear colorless to pale yellow supernatant. The precipitate was entirely composed of inorganic ash. The supernatant contained small amounts of dissolved organic matter and inorganic salts.
5. The gas and vapors were relatively free of contaminants (small amounts of acetone vapor, carbon monoxide, hydrogen were detected).
6. Slurry feed solids concentration was not an important variable in the completeness of oxidation.
7. Continuous stirring of the slurry enhanced oxidation.
8. Temperatures from  $560^{\circ}\text{K}$  ( $550^{\circ}\text{F}$ ) to  $588^{\circ}\text{K}$  ( $600^{\circ}\text{F}$ ) were preferred.
9. Significant quantities of ammonia were found in the effluent water.

Based on these results, a hardware development program was begun to design, fabricate, and test a laboratory prototype wet oxidation system appropriate to spacecraft manned missions (Contract NAS 1-9183). The first step in the development effort was a laboratory investigation to further study the effects of temperature, time, oxygen partial pressure, percent excess oxygen, and solids concentration prior to detail design of the more important components.

A series of 34 batch tests were conducted using fecal/urine slurries in a one-liter stirred reactor and the results indicated that the following process conditions were optimum:

Reaction temperature	560°K (550°F)
Oxygen partial pressure	7.6 MN/m <sup>2</sup> (1100 psi)
Reactor total pressure	15.2 MN/m <sup>2</sup> (2200 psi)
Waste processing time	1-1/2 hours

Slurry solids concentrations of 1.7 to 30.8% were tested and the oxygen required was found to be 0.8 gm oxygen/gm solids. Catalyst tests were also run, but no catalysts were found that would improve process conditions. A test was also run with a mixture of paper towels, toilet paper, wet wipes, aluminum food bags, and photographic film. A high degree of oxidation occurred with the waste materials tested.

Subsequently, the laboratory prototype system was designed, and the components of the design that were essential to the characterization of the process for spacecraft application were fabricated, and tested. The design was based on the results of the laboratory program. The system consisted of an oxygen supply tank, oxygen flow controls, slurry feed tanks and controls, a continuous flow stirred reactor, pressure controls, dry boiler, and miscellaneous controls and gages. Slurry and oxygen were forced into the reactor at controlled rates under pressure. The reactor was designed to stir the mixture and force it through an internal baffling system so that the mixture passed from the inlet to the outlet in 1-1/2 hours, minimizing the mixing of influent and effluent streams. A magnetic coupling with external electric motor drove the stirring mechanism to eliminate a dynamic shaft seal. The reaction products (water, gases, and ash) were vented from the reactor through a cooling coil and back pressure control valve into the dry boiler. The water was sprayed on a hot finned plate to vaporize the water and leaving the salt and ash adhering to the plate. The water vapor passed through a membrane to prevent carryover of any solid particles before entering a condenser where the product water was collected for analysis.

\*Range tested - Process can probably handle any solids concentration that is still fluid.

A 100-hour design verification test (DVT) was run on the system. Results were generally favorable, but a number of areas for further development were identified which formed the basis for the additional work conducted after completion of the DVT. The work accomplished in each work area is summarized briefly below:

**Slurry Pump** - The low flow rates, slurry mixtures, and high pressures made it difficult to locate a slurry pump for the system. Two pumping systems have been developed, one using a motor driven hydraulic piston pump that pumps water to the back side of a bladdered tank to force slurry out of the tank into the reactor, and a second that utilizes the reactor effluent liquid and gas to force the slurry into the reactor using a double-ended slide valve piston pump. The slide valves operate similarly to a steam locomotive slide valve.

**Materials Corrosion** - The requirement for a suitable metal for the reactor and other high temperature portions of the system necessitated a materials test program that exposed metal samples to the wet oxidation environment for extended periods. Hastelloy C was selected as the preferred metal with Inconel 625 a close second choice.

**Oxygen Supply** - Trade-off studies compared high pressure gas, super-critical cryogenic, chemical, and high pressure water electrolysis oxygen supply techniques. Water electrolysis was tentatively selected.

**Water Recovery Devices** - Electrodialysis, vapor compression distillation, vapor diffusion, reverse osmosis, and air evaporation water reclamation systems were compared for the cleanup of the wet oxidation reactor effluent water as alternates to the dry boiler. The trade study resulted in selection of vapor compression as a first choice and electrodialysis as a second choice. Concern about the zero gravity aspects of the dry boiler resulted in elimination of it as a candidate.

**Ammonia Removal** - Chemical analysis of the filtered reactor effluent water that had been boiled and condensed to simulate the vapor compression distillation process showed ammonia as the only contaminant. Its appearance in large quantities (0.55% by weight) presented an important development problem. Surveys of ammonia removal methods resulted in emphasis on finding a catalyst that could be added to the wet oxidation reactor that would prevent ammonia formation. Tests in the one liter batch type laboratory reactor resulted in selection of Ruthenium Trichloride as a very effective catalyst for this purpose that, when introduced in small quantities, completely eliminated the ammonia formation.

Dry Waste Pulverator - A device was investigated that could pulverize the personal hygiene, medical, food, and other biologically contaminated wastes onboard a spacecraft so that they could be pumped into a wet oxidation reactor. A laboratory model was built and tested and design recommendations for a flight prototype were evolved.

Reactor - The DVT showed areas for improvement in reactor design to greatly enhance assembly and disassembly techniques as well as to improve bearing life and drive motor performance. Reactor re-design, a 40-day carbon bearing life test, and a 90-day ball bearing life test were conducted. The carbon bearings failed whereas the ball bearings were still operating at the conclusion of the 90-day test. This test also served as a final checkout of the new system design.

Valves and Regulator - The nature of the sewage with solid particles including fine fibrous material and the finely divided effluent ash coupled with the high pressure requirement made it very difficult to find valves and regulators that would meet requirements. After testing several models, satisfactory components were found.

The laboratory test program, initial prototype testing and subsequent component development effort laid the foundation for the work covered by this report. This step in the development program was initially aimed to design, fabricate and test an engineering prototype wet oxidation system including the improved component designs and system elements indicated by the various tests previously conducted. The priority of the system designs should the complete effort not be economically possible was: 1) reactor subsystem; 2) product recovery subsystem; 3) input subsystem. The latter was further prioritized into: 1) catalyst injection subsystem; 2) pulverator subsystem; 3) high pressure pump; 4) the holding, dispensing equipment for the pulverized slurry; and 5) the oxygen generation system. The product recovery subsystem did not at that time include the goal of providing potable water.

## PROCESS CONDITIONS SELECTION

The wet oxidation process conditions of temperature, pressure, and reaction time selected as a result of the laboratory test program were 560°K (550°F), 15.2 MN/m<sup>2</sup> (2200 psig), and 1 1/2 hours, respectively. These conditions had been selected before the benefits of a catalyst were demonstrated and were used in testing the reactor bearings, slurry pump, and reactor materials in the later phases of Contract NAS 1-9183. Concurrent with these developments, Ruthenium Trichloride (RuCl<sub>3</sub>) was demonstrated to not only suppress ammonia formation, but to also be a good oxidation catalyst. Consequently, one of the first tasks in the new effort was to evaluate the effects of the RuCl<sub>3</sub> on the process conditions.

Process conditions with the catalyst were investigated with the same equipment as the initial tests. A one liter, stirred, batch reactor was charged with 460 cc of fecal/urine slurry (10% by weight feces) and 0.1 gm of RuCl<sub>3</sub>. Oxygen was added to bring the pressure to 5.5 MN/m<sup>2</sup> (800 psig). The vessel was heated to operating temperature with the agitator rotating at 1200 RPM. The vessel was maintained at temperature for the prescribed operating time. The heater was then de-energized and air was blown over the vessel to reduce cool-down time. After cool-down, the vessel was vented, opened, and the liquid removed for analysis. The reactor effluent water was boiled and condensed to simulate processing by a vapor compression distillation unit and the condensate was analyzed on a Perkin Elmer 521 Infrared Spectrophotometer.

The tests showed that process temperature could be reduced to 532°K (500°F) with a corresponding reduction in pressure to 11 MN/m<sup>2</sup> (1600 psig), or process time could be reduced to 0.5 hours if 560°K (550°F) and 15.2 MN/m<sup>2</sup> (2200 psig) were maintained. The system volume, weight and power consumption were more affected by contact time than temperature and pressure, so the process conditions of 560°K (550°F), 15.2 MN/m<sup>2</sup> (2200 psig) and 0.5 hours were selected.

## SYSTEM REQUIREMENTS

The engineering prototype system was to be designed to meet the following requirements:

### Mission Model

Mission Duration	2 years
Resupply Capability	180 days
Gravity Mode	0 to 1 g
Mission Objective	Space Station/Space Base

### Vehicle Model

Compartment Size	3.9 m (156") Diameter 2 m (82") Height
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### Crew Model

Crew Size	6 men
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### Atmosphere Model

Cabin Total Pressure	48 kN/m <sup>2</sup> (7.0 psig) to 102 kN/m <sup>2</sup> (14.7 psia)
Gas Composition	24 kN/m <sup>2</sup> (3.5 psia) O <sub>2</sub> , Balance N <sub>2</sub>
Carbon Dioxide Partial Pressure	0 to 0.4 kN/m <sup>2</sup> (3 mmHg)
Temperature	291° (65°F) to 297°K (75°F)

### Waste Model

	<u>Solids</u> <u>kgm/man-day (lb/man-day)</u>		<u>Liquid</u> <u>kgm/man-day (lb/man-day)</u>	
Urine	0.59	(0.13)	1.5	(3.31)
Feces	0.032	(0.07)	0.09	(0.20)
Waste Flush Water			2.4	(5.30)
Food Wastes	0.0454	(0.10)	0.136	(0.30)
Wipes	0.0450	(0.10)	0.0454	(0.10)
Housekeeping, Hygiene	.068	(0.15)	-	-
	0.25	(0.55)	4.18	(9.21)

Total 6 man load      27.8 kgm/day (61.3) lb/day

### System Function

The system design goal was to provide the following functions:

- o Pulverize spacecraft wastes other than fecal/urine wastes to provide a pumpable slurry and the transfer of these wastes to a hold tank.
- o Pump the waste slurry to reactor pressure and deliver the slurry to the reactor.
- o Introduce oxygen at the design rate into the reactor feed system.
- o Heat the input slurry/oxygen mixture to near reactor temperature using heat from the effluent liquid and gases.
- o Oxidize the wastes in a stirred reactor in which the residence time was planned to be 0.5 hr.
- o Control the system pressure and the venting of the effluent liquid and gases.
- o Filter the effluent to remove ash produced by the oxidation process.
- o Separate the liquid and gas effluents.
- o Monitor and control system functions to provide safe system operation automatically, and system shutdown to a safe condition automatically.



## PRELIMINARY DESIGN

Although, the previous developments demonstrated many features of component designs and the optimum process conditions, a number of problems remained to be resolved in a preliminary design phase. The most significant of these were:

- 1) Establishing a means of introducing 1.0 gm of  $\text{RuCl}_3$  for every 4600 cc of slurry.
- 2) Establishing a means of introducing 5 kgm per day (11 lb/day) of oxygen into the system.
- 3) Sizing influent and effluent tanks.
- 4) Selection of a slurry pump from the two primary candidates (hydraulic piston and slide valve piston).
- 5) Optimization of the regenerative heat exchanger.
- 6) Selection of a reactor bearing design and stirring system arrangement.
- 7) Comparison of several alternate filter concepts.
- 8) Comparison of phase separator concepts.
- 9) Selection of a pulverizer concept.
- 10) Selection of motorized ball valve designs.

### Catalyst Introduction System Comparisons

The amount of catalyst required to prevent ammonia formation in the wet oxidation of spacecraft wastes was established during the laboratory test program to be approximately 0.1 gm of  $\text{RuCl}_3$  for 460 cc of slurry. Based on this requirement and a specified 27.8 kgm/day (61.3 lb/day), of slurry, 6.1 gm per day of  $\text{RuCl}_3$  was required. Measurements showed 1 gm of  $\text{RuCl}_3$  in 4 gm of  $\text{H}_2\text{O}$  produced 4.3 cc of solution. The catalyst flow rate was calculated to be 6.1 (4.3) or 26.2 cc/day (1.1 cc/hr or 0.018 cc/min). The catalyst supply tank volume for a 90 day test period was calculated to be  $26.2 (90) = 2360$  cc or 145 in<sup>3</sup>.

Based on these requirements a number of catalyst introduction schemes presented by Table 1, were evolved and compared. All of the systems listed are based on injecting the catalyst into the low pressure slurry feed, or hold system, except number 6 which feeds the catalyst into the high pressure portion of the system. All of the systems have their drawbacks. Number 3 was the preferred approach if a low flow metering pump were available. Number 5 was selected as a best available technology compromise. Figure 2 presents a schematic of the selected catalyst introduction system. Slurry from the hold tank is continuously pumped into the reactor. Catalyst from the supply tank is forced through a filter, capillary tube bundle and solenoid shut-off valve into the suction side of the slurry pump. A constant  $\Delta P$  is maintained across the capillary tubes by gas regulators on the catalyst supply tank and the slurry hold tank.

#### Oxygen Introduction System

Oxygen required for the 27.8 kgm/day of wastes was calculated by several methods giving quite a wide range of results based on the assumed chemical composition of the waste model. Requirements ranged from 2.74 kgm to 5.18 kgm per day (6-11.4 lb/day). It was decided to base the system design on a maximum flow of 5.18 kgm per day and be capable of varying it down to 1.8 kgm per day. This produced a requirement of 53 to 150 standard liters per day.

Two concepts of flow control were considered. One utilized a sintered metal disc as a flow orifice with flow varying with  $\Delta P$  across the orifice and the other utilized a close tube filled from a higher pressure source and periodically dumped into the reactor feed line by pulsing a three way solenoid. The metal disc orifice approach was selected as being the simplest, most reliable method. Figure 3 presents a schematic of the selected system. Oxygen from a supply source at  $16.6 \text{ MN/m}^2$  (2400 psig) or greater is delivered to the oxygen regulator that maintains  $16.6 \text{ MN/m}^2$  (2400 psig) on the upstream side of the orifice. A  $5 \mu\text{m}$  filter protects the orifice from clogging. The orifice discharges the oxygen into the reactor feed line through a solenoid operated shut-off valve. Reactor pressure is maintained at  $15.2 \text{ MN/m}^2$  (2200 psig) by a back pressure regulator in the reactor effluent line.

Table 1 Catalyst Introduction Systems

<u>Candidate Systems</u>	<u>Remarks</u>
1. Pellets injected into input slurry hold tank.	o Assures precise quantity but injection mechanism is extremely complicated.
2. Solid rod located in or introduced into slurry hold tank.	o Complicated injection system and dissolving rate difficult to control.
3. Liquid solution pumped into slurry feed system (low pressure).	o Difficulty in locating pump for such low flows ( $\approx 1$ cc/hr.)
4. Constant $\Delta P$ across a porous membrane.	o Possible clogging of membrane.
5. Constant $\Delta P$ across a capillary tube bundle.	o Possible clogging of capillary tubes.
6. Liquid solution pumped into reactor system (high pressure).	o Difficulty in locating pump for such low flows particularly at high pressures ( $\approx 1$ cc/hr at 2200 psi). Provides the most uniform flow distribution.

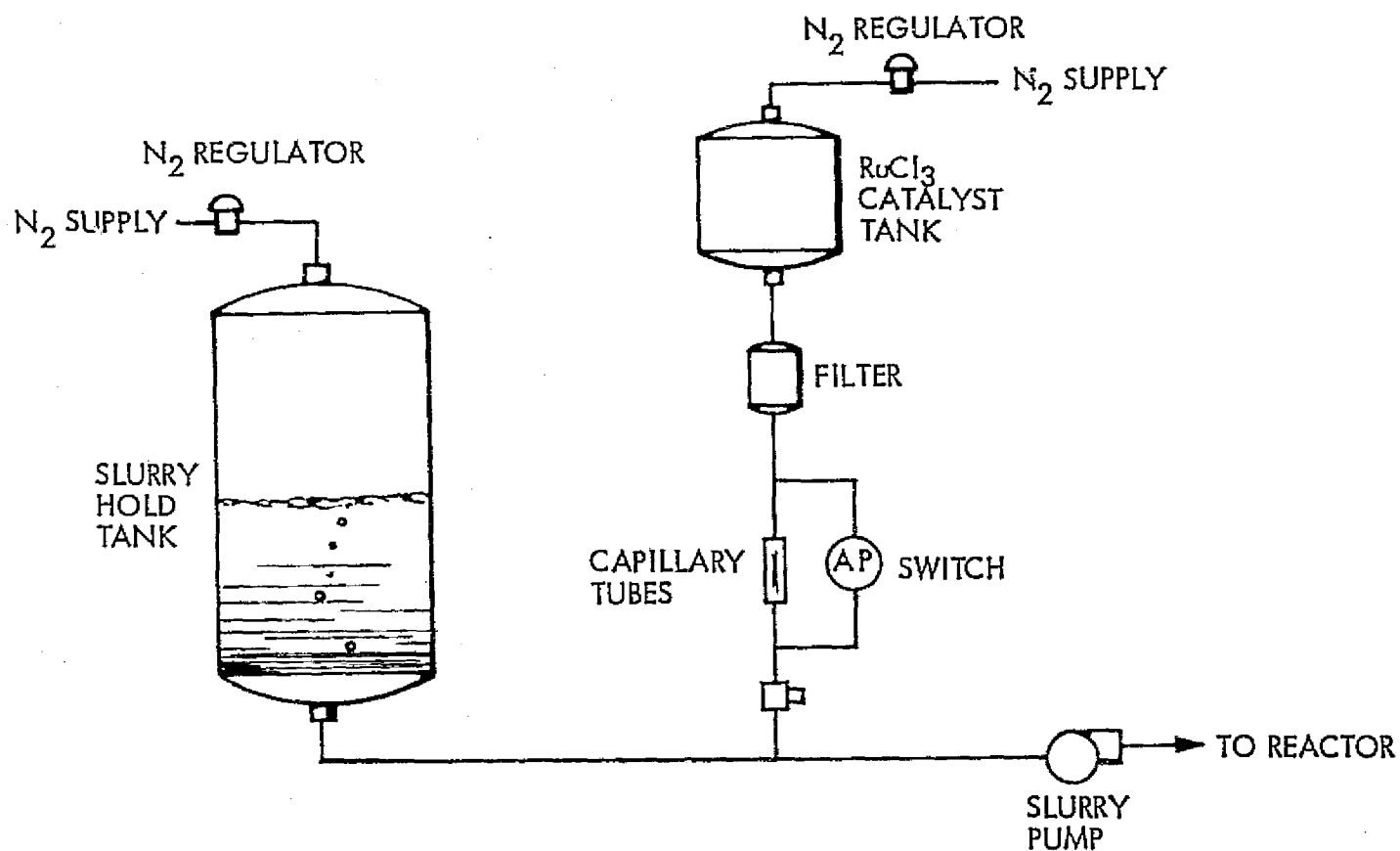


Fig. 2 Selected Catalyst Introduction System

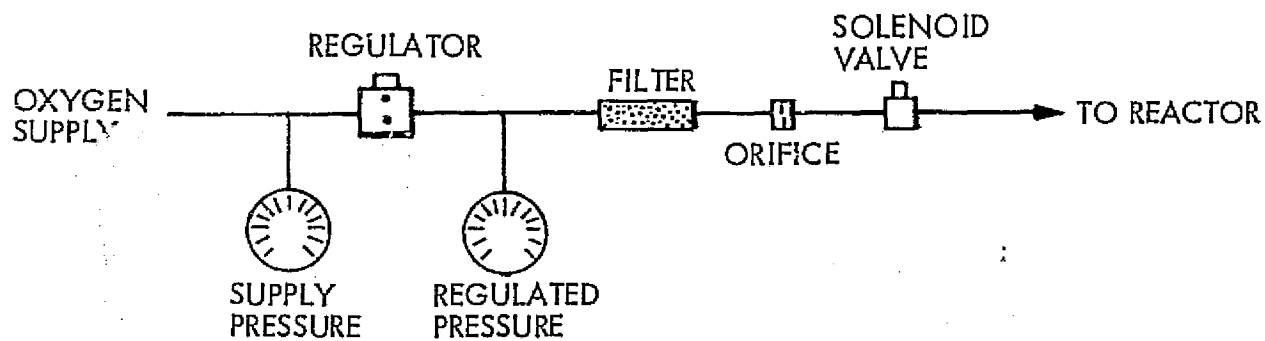


Fig. 3 Selected Oxygen Introduction System

## Slurry & Effluent Tank Sizing Study

A study was made to establish the size and design concept for the input slurry and effluent water hold tanks. Reference 2 was used as a source of information concerning crew activities and waste generation rates. The study was conducted by establishing the waste generation rate during a typical day, plotting the accumulation versus time, and subtracting the amount processed versus time; which produced the curve presented by Figure 4. The curves are presented (1) for nominal load conditions, (2) for maximum load condition and (3) minimum waste generation based on complete shutdown of the system and the crew on emergency rations. Tank capacity must be 9.76 kgm for the maximum design load plus 7.49 kgm for a 24 hour shutdown assuming the shutdown occurred immediately after peak loading at 0800 hours. This results in a 18.25 kgm total slurry hold tank capacity. A tank with total volume of 1.9 MM<sup>3</sup> (5 gallons) would be adequate. The effluent water tank size can be minimal because if a failure occurred in the downstream processing system the wet oxidation slurry hold could be used to accumulate wastes until the water processor could be repaired. A 0.76 mm<sup>3</sup> (2 gallon) effluent water tank was selected to provide approximate 6 hours of effluent water capacity under normal operating conditions and 24 hours under emergency conditions. Figure 5 presents the tank design selected for the slurry and effluent water storage. The metal bellows provides positive expulsion and allows volume indication for control purposes.

## Slurry Pump Comparison

Pumping of the waste slurry from spacecabin pressures to reactor pressure 15.2 kn/m<sup>2</sup> (2200 psi) at a constant, very small flowrate was recognized early in the development program to be a significant problem. Two pumping approaches were evolved and tested in contract NAS 1-9183. The hydraulic piston pump presented by Figure 6 utilizes a small bore motor driven piston to pump water to the back side of a bladdered tank full of slurry. The slurry is forced out of the tank at

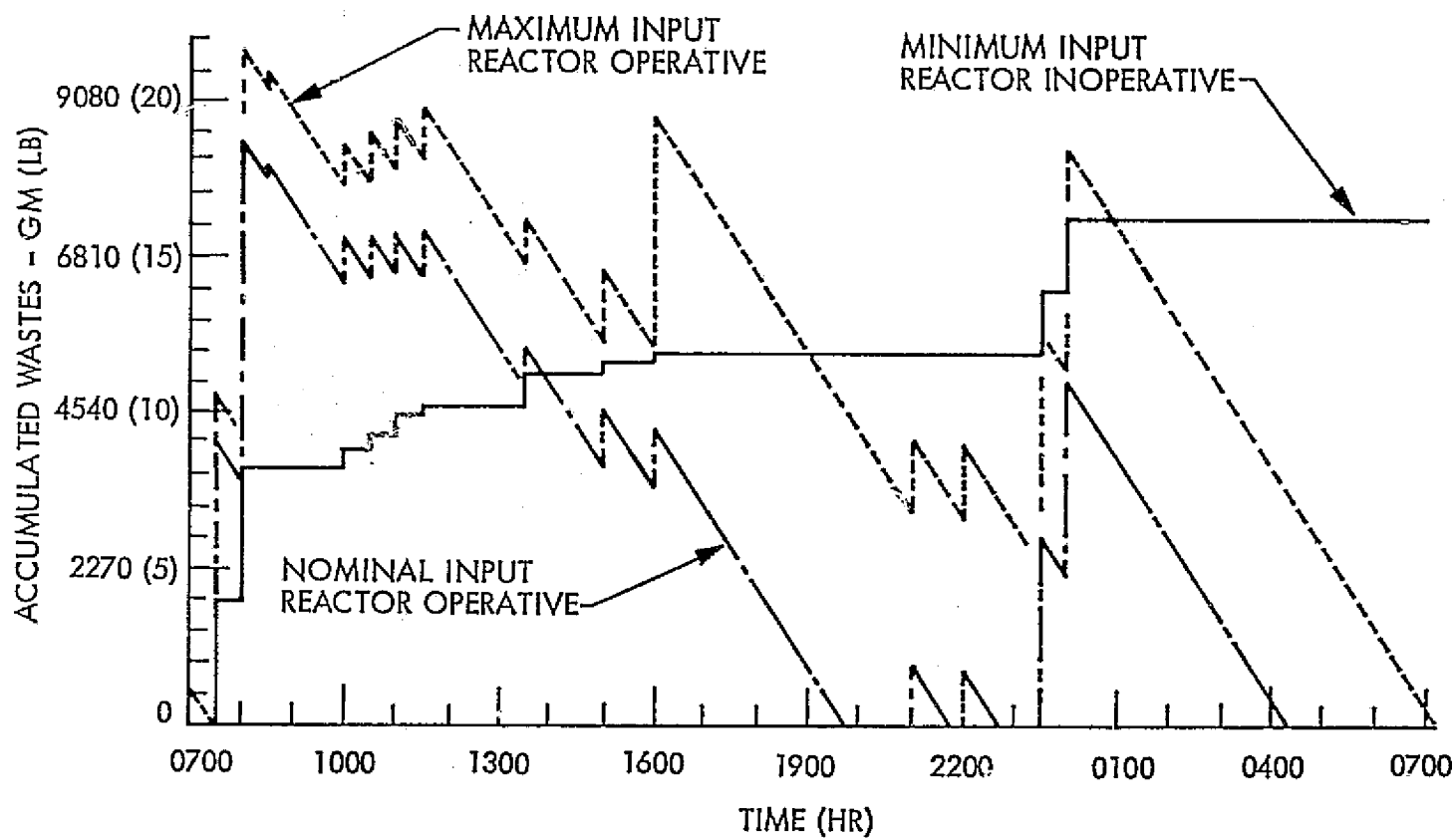


Fig. 4 Slurry Accumulation for a One Day Cycle

	SLURRY TANK	EFFLUENT TANK
VOLUME LITERS (GAL)	18.9 (5)	7.6 (2)
MATERIAL	CRES	CRES
VOLUME MEASURE- MENT	FULL & EMPTY	FULL & EMPTY
MAXIMUM PRESSURE KN/M <sup>2</sup> (PSI)	345 (50)	345 (50)
P/N	E63286-2	E63286-1
MFG	METAL BELLOWS CORP.	METAL BELLOWS CORP.

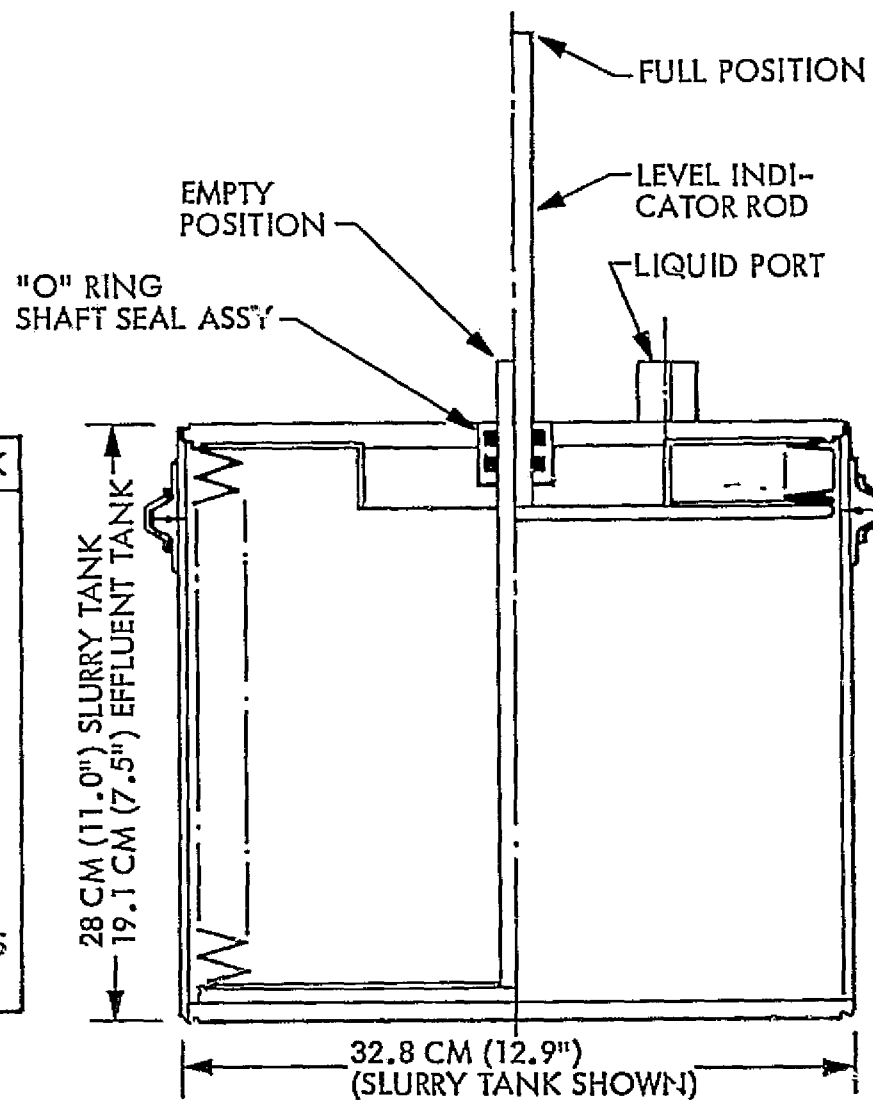
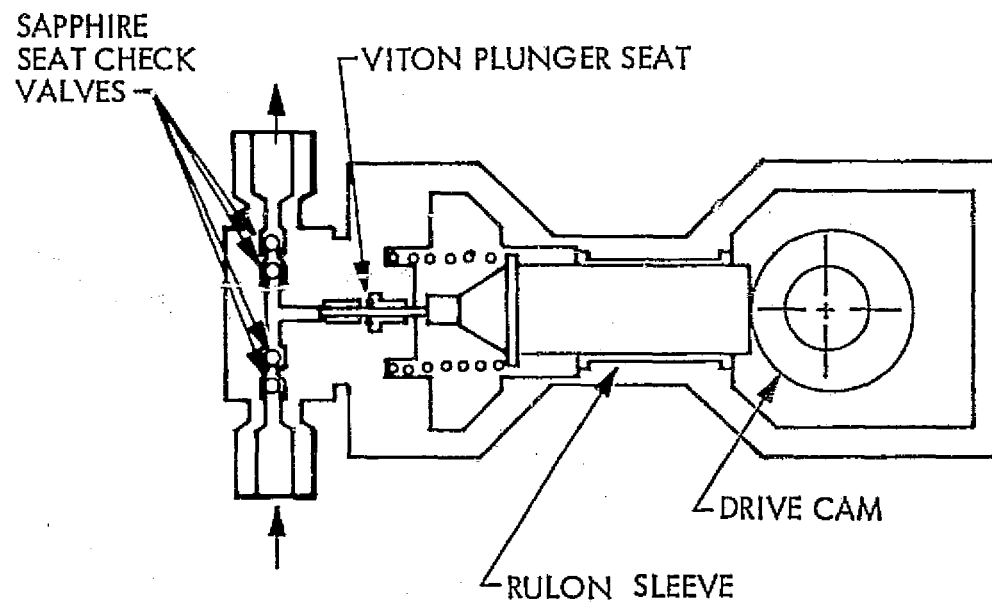


Fig. 5 Slurry &amp; Effluent Tank Description





MILTON ROY CO. HIGH PRESSURE MINIPUMP  
ONE OF TWO PUMP HEADS SHOWN

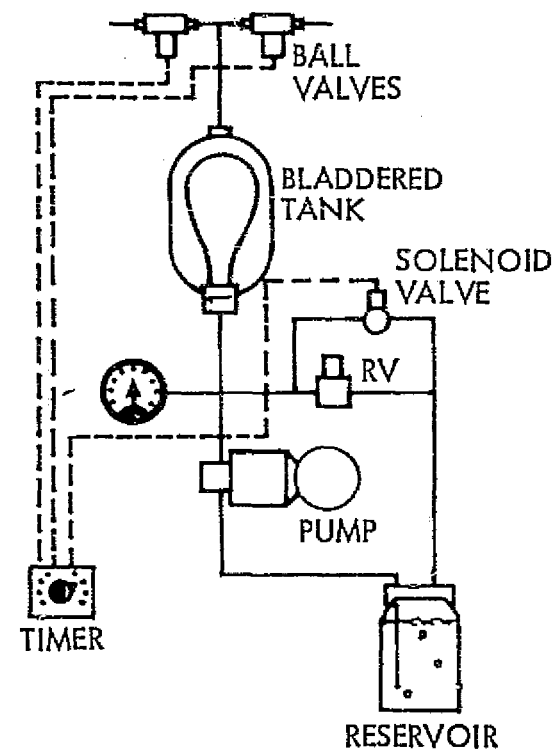


Fig. 6 Hydraulic Pump Configuration

a predictable rate by the positive displacement pump. A timer controls the operation of the two slurry ball valves and the hydraulic pump bypass solenoid to allow (1) filling of the bladdered tank with slurry, (2) pressurization of the tank to reactor pressure using the hydraulic pump and (3) delivery of the slurry to the reactor at a steady selectable rate. The hydraulic pump is commercially available, so this system offered the advantage of being state-of-the-art and required little development effort. The primary disadvantage is the relatively high weight and number of parts required.

The double-ended, slide valve, slurry pump presented by Figure 7 was designed, fabricated, and tested under contract NAS 1-9183. It is a free floating piston pump using ported slide valves. The free floating piston is forced down by slurry from the hold tank and is forced up by the effluent from the reactor. The slide valves were pushed back and forth by hydraulic actuators on the original test unit, but would be driven by an electric motor and drive train on a flight version. The pump delivers slurry to the reactor while oxygen for the process is metered by separate controls. Both liquid and gas effluents from the reactor are used to drive the pump, thereby providing energy to make up for friction and pump inefficiency.

The slide valve slurry pump offers the advantage of fewer components and lighter weight, but requires development and complicates the effluent processing system. The effluent is discharged through a filter and phase separator into an effluent water tank. In order to prevent large pressure surges in these components as the slurry pump discharges high pressure gas, a relatively large accumulator is needed.

The characteristics of these two pumping approaches are compared in Table 2. The hydraulic pump was selected because it required less development effort and was therefore more suitable for use in the demonstration system where emphasis was being placed on overall system integration and operation and the reactor and product recovery subsystems.

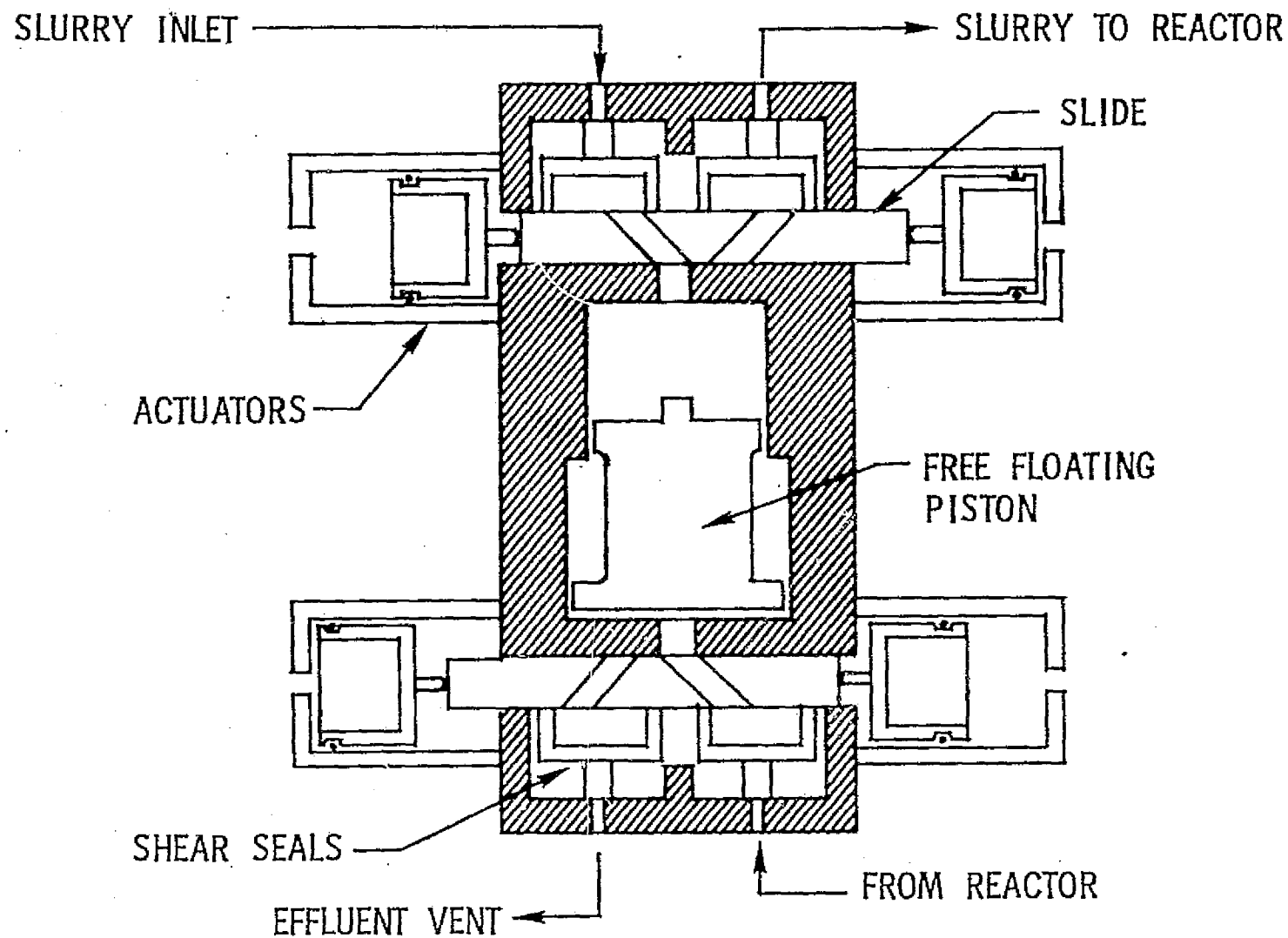


Fig. 7 Slide Valve Pump Configuration

Table 2 Slurry Pumping Comparison

Requirements	Comparison	Hydraulic Pump	Slide Pump
Slurry Flowrate, 27.9 kgm/day (61.4 lb/day)	Total Effective Weight kgm (lb)	23.15 (51.0)	16.98 (37.4)
Slurry Temperature, 298°K (75°F)	Volume m <sup>3</sup> (ft <sup>3</sup> )	.051 (1.8)	.096 (3.4)
Suction Pressure, 102 kN/m <sup>2</sup> (15 psia)	Power, watt	19.5	6.5
Delivered Pressure 15.2 MN/m <sup>2</sup> (2200 psia)	Number of Components (Spares Included)	12	3
Continuous Duty, 180 days			
SS Construction	Reliability	High	High
	Filter	Every 15 Days	Every 3 Days

## Regenerative Heat Exchanger Design Study

The early conceptual designs of a spacecraft wet oxidation system included a regenerative heat exchanger to heat the incoming slurry and oxygen using the hot liquid and gases discharging from the reactor. The earlier test system did not include a heat exchanger, however, so a tradeoff study was conducted to select the heat exchanger design and optimize the configuration. Figure 8 presents three candidate heat exchanger designs. The side by side tube configuration was eliminated because of poor heat transfer characteristics through the connecting metal. The plate and fin design was not practical for high pressure applications and would have been very expensive to fabricate. The tube in tube design was selected.

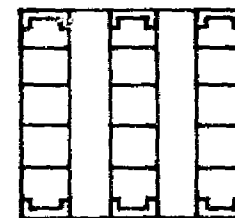
Figure 9 presents the results of a heat exchanger optimization study which calculated tube length and total equivalent weight as a function of heat exchanger effectiveness. The combined two phase flow including evaporation, made calculation of the heat transfer coefficient impractical. Hence, the tube length needed for a counterflow, tube in tube heat exchanger was calculated by estimating the energy required to heat the slurry/oxygen mixture to the heat exchanger outlet temperature and dividing by an estimated  $\Delta T$ , tube area per linear foot and overall heat transfer coefficient. The tube configuration was selected from empirical information available from a previous program. The inside tube was .95 cm (3/8") with 0.71 mm (.028") tube wall inside of a 1.59 cm (0.625") tube with 10.7 mm (.421") tube wall. The overall heat transfer coefficient was estimated to be 1700 watts/hr, m<sup>2</sup>, °K (300 BTU/hr-ft<sup>2</sup> - °F). The total equivalent weight was derived from the tube length and included the weight of the heat exchanger tubes and tube supports, insulation, cover, and weight equivalent for the heat leak and power required to heat the slurry/oxygen mixture from heat exchanger outlet temperatures to reactor temperature. Figure 9 shows an optimum tube length of 12 feet at a heat exchanger effectiveness of 80 percent.

Figure 10 shows the preliminary design of the regenerative heat exchanger based on the results of the optimization study. It was assumed that the tubes could be bent to a minimum radius of 1 times the outside tube diameter. This resulted in a heat exchanger size of 11.7 cm (4.6") x 37.6 cm (14.8") x 45.7 cm (18.0") including 5.1 cm (2 inches) of foam insulation.

## COMPARISON

SIDE BY SIDE  
TUBES

## TUBE IN TUBE

COMPACT HEAT  
EXCHANGER

- TOTAL EFFECTIVE WEIGHT
- VOLUME
- COST
- COMPLEXITY
- EASE OF FABRICATION
- DIFFERENTIAL THERMAL EXPANSION

HIGH

HIGHEST

LOW

LOW

NOT DIFFICULT

MODERATE

LOW

MODERATE

LOW

LOW

NOT DIFFICULT

SELF RELIEVING

MODERATE

LOWEST

HIGH

MODERATE

MODERATELY  
DIFFICULT

LOW

Fig. 8 Regenerative Heat Exchanger Candidates

# ASSUMPTIONS

- 1 REACTOR PRESSURE 15.2 MN/M<sup>2</sup> (2200 PSIA)
- 2 REACTOR TEMPERATURE, 560°K (550°F)
- 3 PROCESS RATE, 27.9 KGM/DAY (61.4 LB/DAY)
- 4 SLURRY TEMPERATURE, 298°K (75°F)
- 5 OXYGEN SUPPLY RATE, 0.8 GMO<sub>2</sub>/GM SOLID
- 6 CABIN TEMPERATURE, 297°K (65°F)
- 7 INSULATION PROPERTIES,  $\rho = 41.4$  KN/M<sup>2</sup> (6.0 LB/FT<sup>3</sup>),  
K = 0.113 WATTS/HR-M<sup>2</sup>-°K (.02 BTU/HR FT<sup>2</sup>-°F)
- 8 CONCENTRIC TUBES, 0.95 CM x 0.071 CM (.375 x .028) AND  
1.59 CM x 0.107 CM (.625 x .042), HASTELLOY C
- 9 AVERAGE INSULATION THICKNESS, 6.08 CM (2.0 INCH)
- 10 POWER PENALTY, 268.3 KGM/KW (591 LB/KW)
- 11 HEAT REJECTION PENALTY, 38.1 GM/K JOULE-HR (0.074 LB/BTU/HR)
- 12 WEIGHT INCLUDES: HEAT EXCHANGER, HEAT EXCHANGER INSULATION,  
HEAT EXCHANGER HEAT LEAK POWER & REJECTION PENALTIES, POWER  
PENALTY TO HEAT SLURRY TO 560°K (550°F)

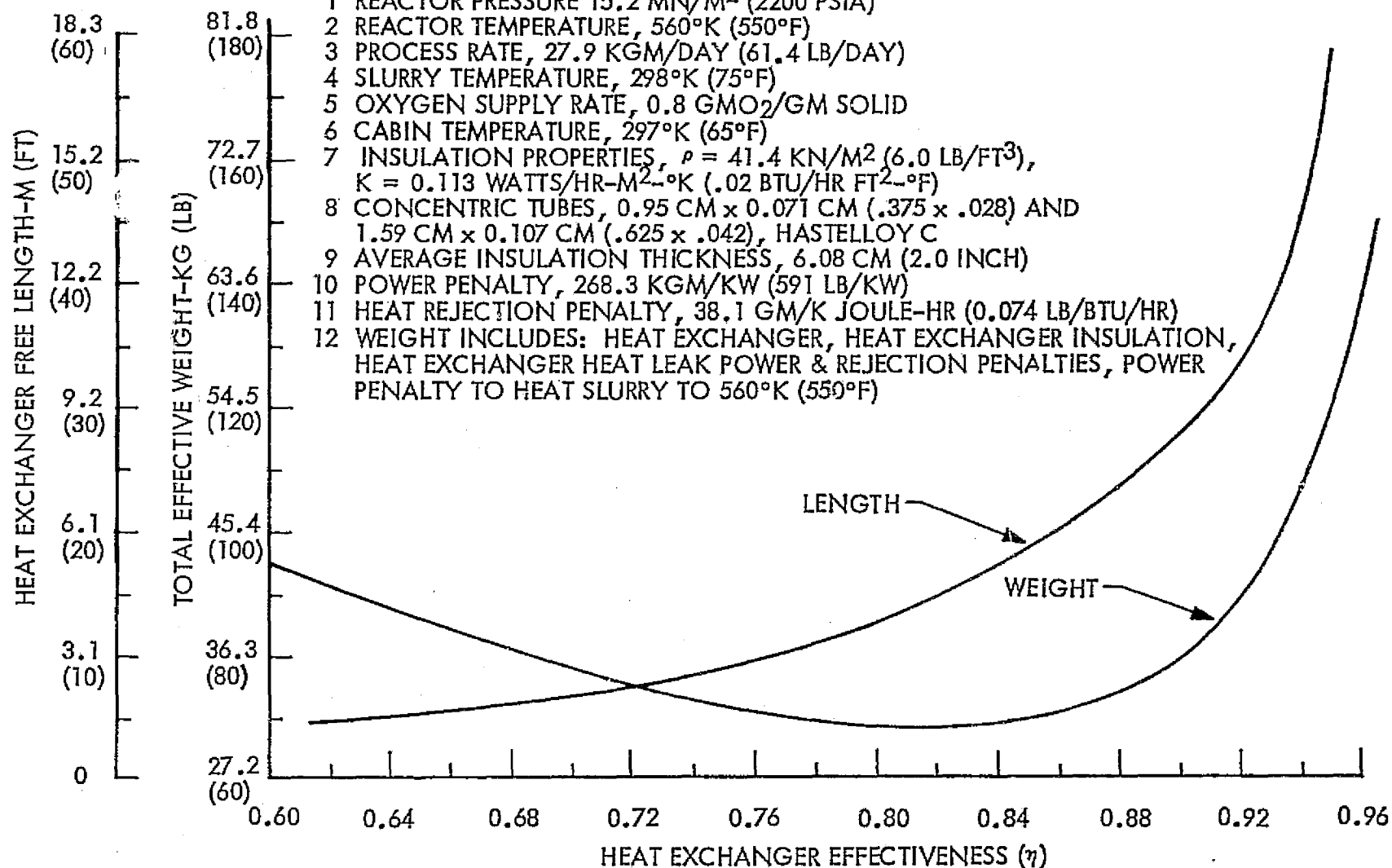


Fig. 9 Heat Exchanger Optimization Results

### SPECIFICATIONS

- EFFECTIVENESS 80 PERCENT
- EFFLUENT OUTLET TEMP 350°K (170°F)
- REACTOR INLET TEMP 514°K (4600°F)
- SLURRY FLOWRATE 1.16 KG/HR (2.56 LB/HR)
- GAS FLOWRATE 0.1 KG/HR (0.23 LB/HR)
- HEAT LEAK 19.7 WATTS/HR (67 BTU/HR)
- WEIGHT 4.26 KG (9.4 LB)
- FREE LENGTH 3.95M (12 FT)
- TUBE SIZE 0.95 CM x 0.071 CM (.375" x .028")  
1.59 CM x 1.07 CM (.625" x .042")
- TUBE MATERIAL HASTALLOY C
- INSULATION FOAM
- OPERATING PRESSURE 15.2 MN/M<sup>2</sup> (2200 PSIA)

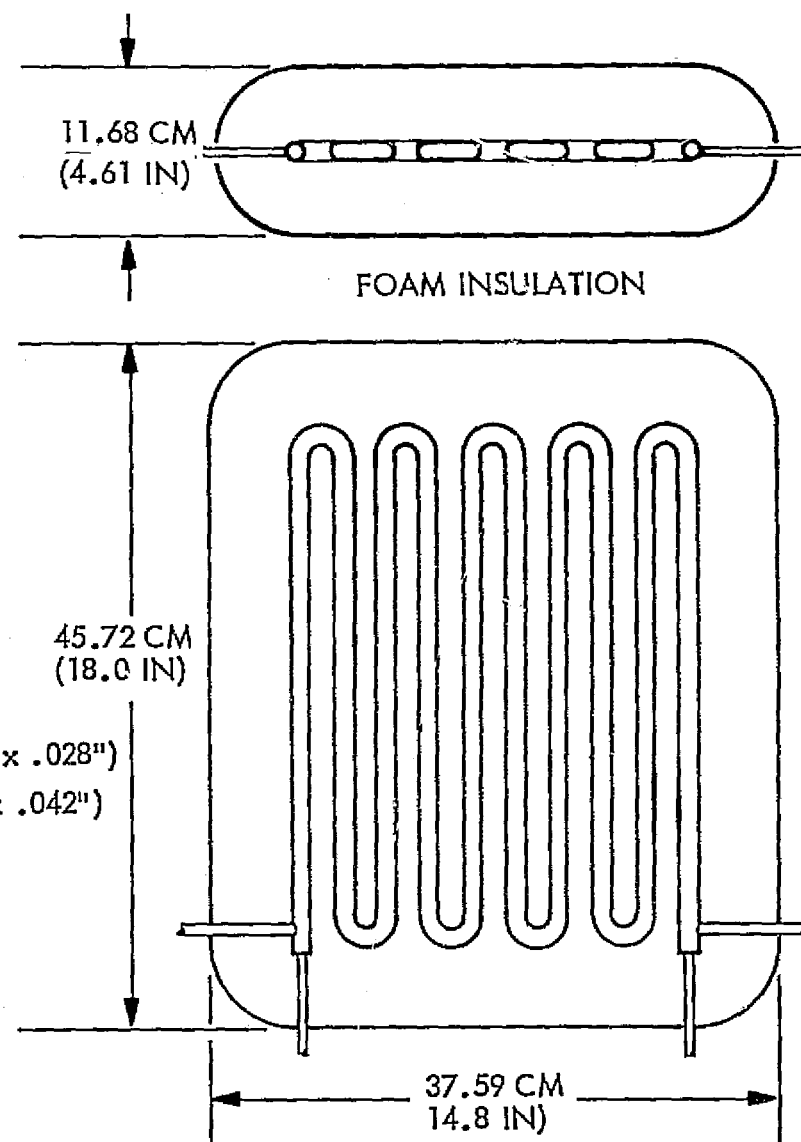


Fig. 10 Regenerative Heat Exchanger Design



## Reactor Design Studies

The reactor design utilized in the final testing during contract NAS 1-9183 is presented in Figure 11. It provided a reactor body, end caps, magnetic drive assembly, internal baffle and dam assembly, stirring shaft, ball bearings, inlet and outlet fittings, heaters, and insulation jacket. A 90 day ball bearing life test conducted in this reactor showed favorable results with all design elements of the system except for two factors. The ball bearings were still functioning after 90 days of testing, but showed signs of severe electrolyte corrosion. The dissimilar metals used in the reactor (Inconel 625) and the bearings (Cobalt-Tungsten alloy) were blamed for the corrosion. The reactor was also very difficult to disassemble for replacement of bearings and end cap seals. In most cases the entire reactor had to be taken apart to replace any part. It was concluded from the preliminary design study that the same basic reactor design using Hastelloy C-276 for all internal reactor parts including ball bearings would be used for the new demonstration test system. A requirement was established that the final design must provide a reactor allowing any bearing or seal to be replaced by pulling only one head and not pulling the baffle assembly.

## Filter Sizing and Selection

Three filter concepts for ash removal from the wet oxidation effluent were studied and are compared by Table 3. Cotton wound depth cartridge, fiber bag, and surface wirecloth filters were compared. The high loading capability, low weight and volume, and low cost resulted in selection of the bag filter as the primary filter. Although previously conducted tests indicated that a single  $5\mu$  m filter would be adequate, there was some concern that a finer polishing filter should be used downstream of the bag filter. A Pall, sintered metal,  $2\mu$  m, filter was selected. Figure 12 describes the two selected filter designs.

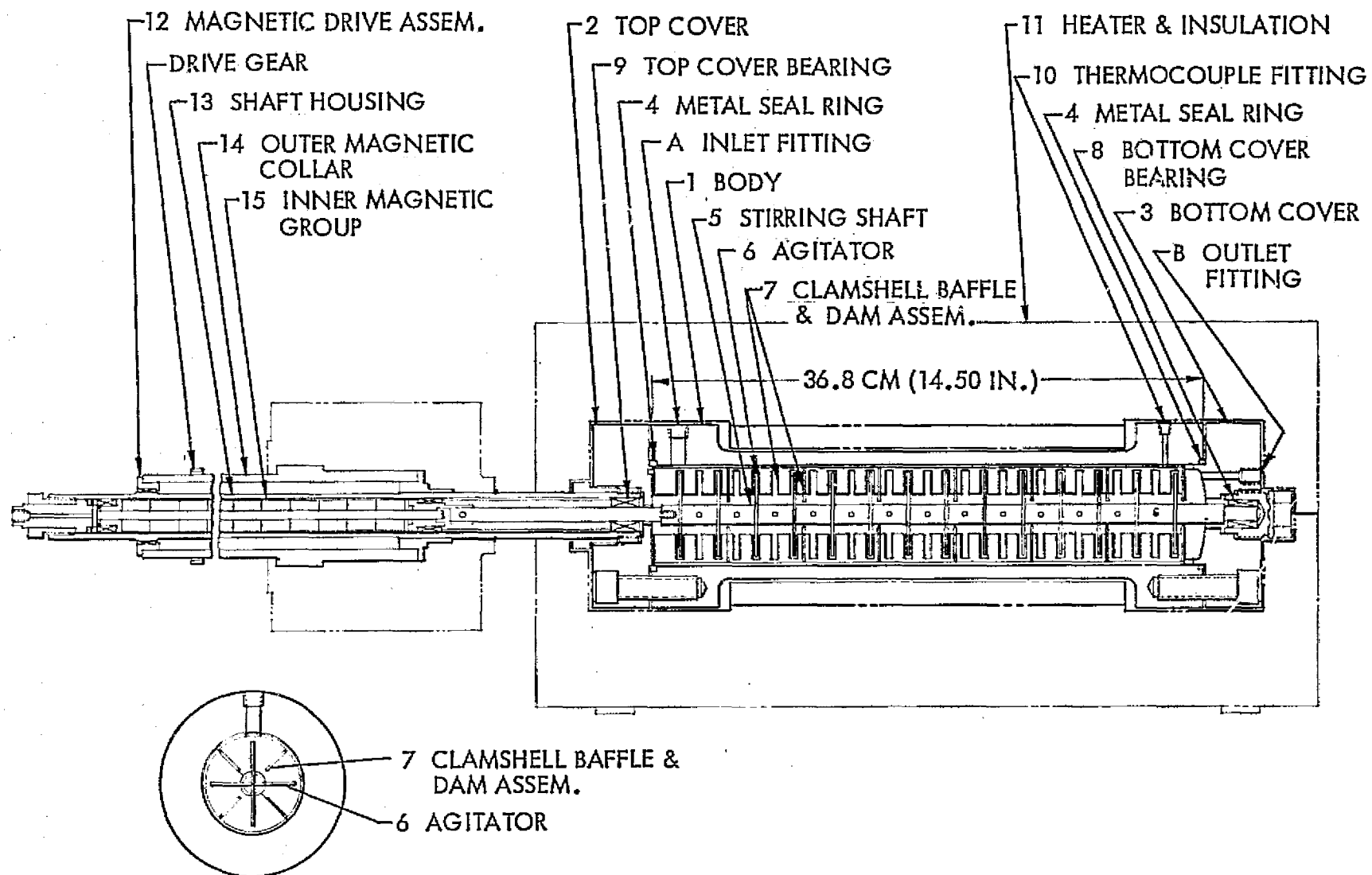


Fig. 11 Reactor Design

Table 3 Filter Candidates

Requirements		Depth Cartridge	Depth Bag	Surface Wirecloth
Particulate Size 0.5 - 1.0 $\mu\text{m}$	o Weight-kgm (lb)	Low 4.54 (10)	Low 3.18 (7)	High 18.16 (40)
Gas Volumetric Flowrate 283 cc/min	o Volume-1 (ft <sup>3</sup> )	36.8 1 (1.3)	255 1 (0.9)	14.2 1 (5)
Liquid Volumetric Flowrate 1200 cc/hr	o Loading Capability	High	Highest	Very Low
Element $\Delta P$ Approx 550 kN/m <sup>2</sup> (80 psia)	o Element Migration	Little	Little	None
Ash Flowrate 2.5 gm/hr	o $\Delta P$ Capability	High	High	High
Ash Density 688 kgm/m <sup>3</sup> (43 lb/ft <sup>3</sup> )	o Recommended Rating ( $\mu\text{m}$ )	5	5	1
Operating Temperature 350°K (170°F)	o Cost	Low	Low	Moderate

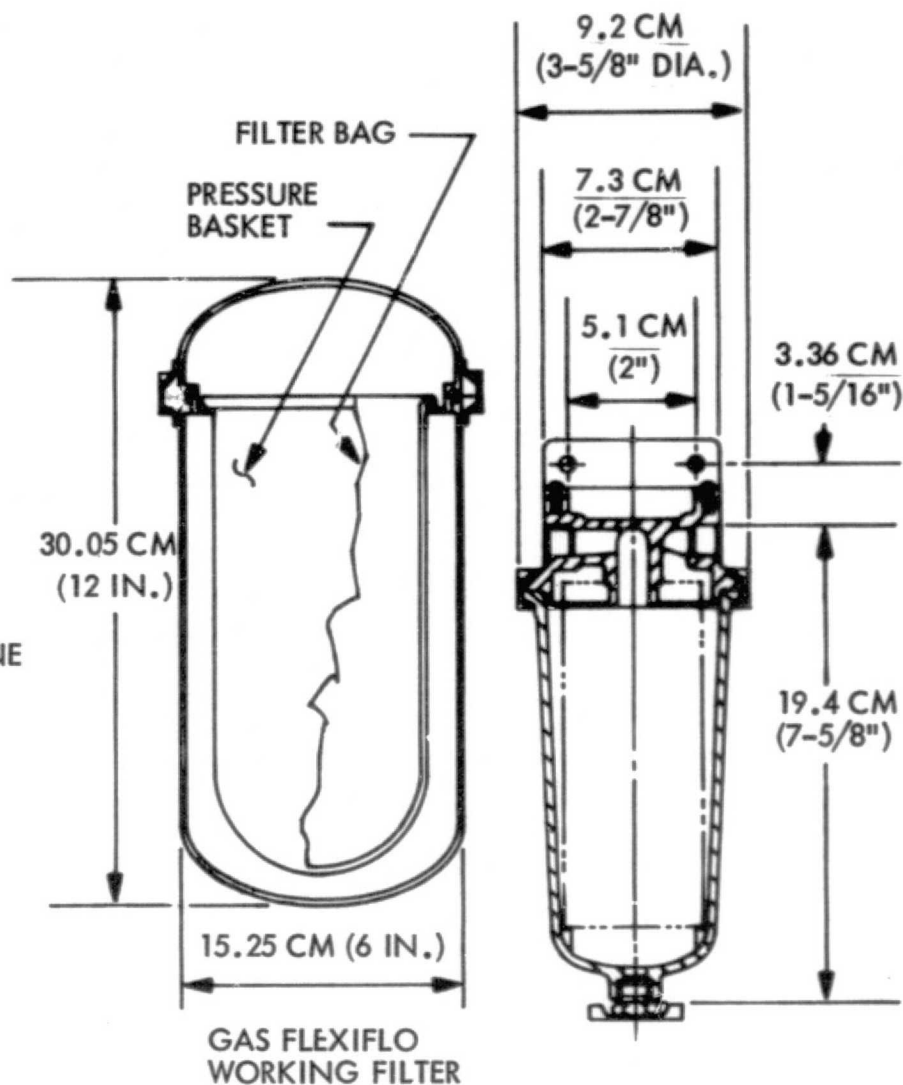
- VOLUME CC (IN.<sup>3</sup>)
- REPLACEMENT FREQUENCY
- RATING ( $\mu$ ) (NOMINAL)
- ELEMENT MATERIAL
- HOUSING MATERIAL
- MAXIMUM  $\Delta P$  ESTIMATED KN/M<sup>2</sup> (PSI)
- MAXIMUM HOUSING PRESSURE KN/M<sup>2</sup> (PSI)

DESCRIPTION

WORKING FILTER

POLISHING FILTER

5900 (360)	1475 (90)
10 DAYS	NONE
5	2
VISCOSE FELT CRES	3161 SINTERED POLYPROPYLENE
518 (75)	518 (75)
863 (125)	863 (60)



PALL TRINITY MICRO CORP.  
CARTRIDGE MCS4463 PH  
POLISHING FILTER

Fig. 12 Selected Filter System

### Phase Separator Comparison

A phase separator is required to separate the liquid and gas effluents of the wet oxidation system downstream of the ash filters. The separator must process 0 to 1200 cc/hr of water and 0 to 1500 cc/min of gas at 20 psig. Two separators developed for use in other spacecraft life support systems were considered for use in the wet oxidation system. Figure 13 presents a schematic drawing of the vortex, centrifugal, phase separator developed for separating gas bubbles from the circulating electrolyte in a zero gravity water electrolysis cell. An electric motor acting through a magnetic coupling drives the vortex, centrifugal pump impeller to provide separation of the liquid and gas phases. A second freely rotating impeller is driven by the rotating liquid at a speed dependent upon the amount of liquid in the pump housing. A speed pickup measures the RPM of the driven impeller and acts through a controller to open or close a gas vent solenoid valve. Gas is vented from the pump housing at the center or eye of the pump housing and liquid is vented at the periphery.

Figure 14 presents a sketch of a hydrophilic phase separator similar in design to one developed for use in a zero gravity, urine, vacuum distillation unit. Gas and liquid are spiraled through the chamber. The liquid strikes the hydrophilic surface and passes through the wetted surface. Gas continues through the spiral to the bottom chamber and out through a tube running through the center of the separator chamber. A slightly reduced pressure is maintained on the liquid outlet side of the hydrophilic surface by a AP switch and water pump.

In comparing the two separators little difference was found in weight, volume, and power, since both utilize water pumps and controls.

The vortex, centrifugal separator was chosen for the wet oxidation system, because it was in a more advanced stage of development and had performed well during the water electrolysis test program.

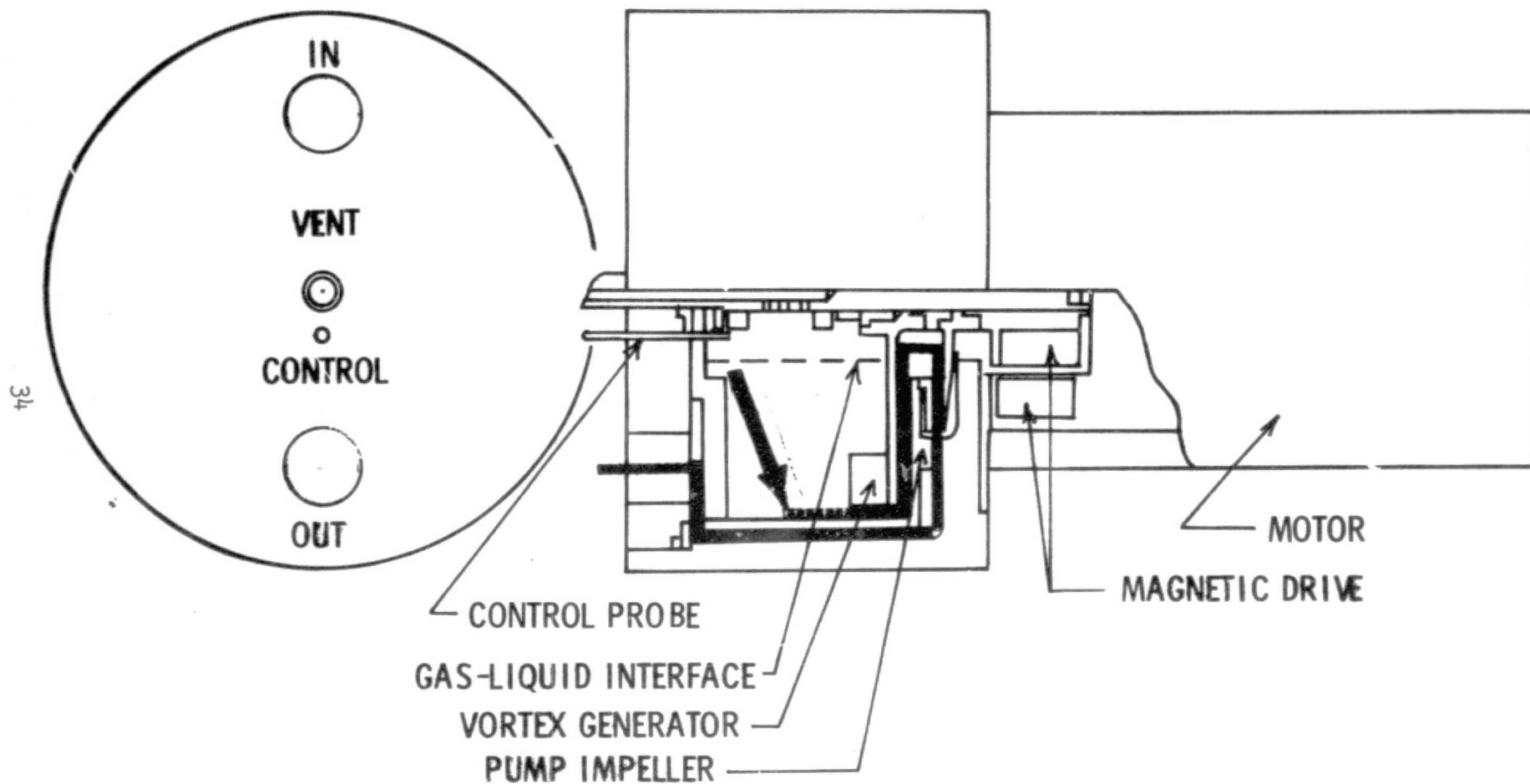


Fig. 13 Vortex Separator and Pump

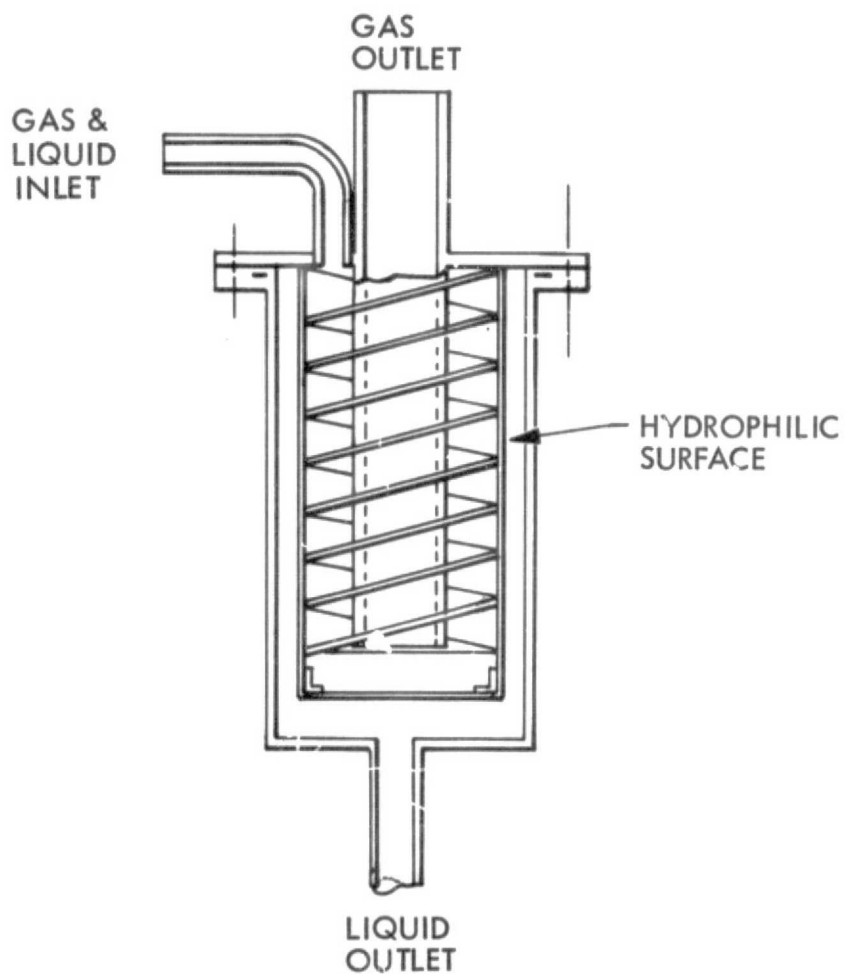


Fig. 14 Hydrophilic Phase Separator

### Dry Waste Pulverizer Subsystem

The laboratory tests run on the waste pulverizer assembled under contract NAS 1-9183 resulted in the conceptual design of a spacecraft pulverizer subsystem shown schematically by Figure 15. The motor driven pulverizer is loaded manually with miscellaneous spacecraft wastes. The ground trash hold tank is half filled with concentrated wash water residue or other waste water. The slurry recycle pump, phase separator and pulverizer are energized. The slurry recycle pump circulates waste water and ground trash from the hold tank back to the pulverizer to assist in pulverizing the trash loaded into the pulverizer. The centrifugal phase separator, pumps the ground trash and water back to the hold tank. The pulverizer is stopped and reloaded as often as required to process the daily waste load. All spacecraft wastes listed in the waste model other than feces, urine and toilet flush water are processed through the pulverizer. When the trash hold tank becomes full the pump is energized and the diverter valve positioned to transfer the ground trash from the pulverizer system into the wet oxidation slurry hold tank.

The preliminary design effort defined the approach to be taken in further developing the trash pulverizer subsystem as part of the demonstration system. The system would consist of a pulverizer designed and fabricated as a zero gravity prototype, connected to a non-zero gravity dropout tank, commercially available recirculation pump and three way valve. The system was to be manually operated. Figure 16 schematically depicts the system planned for the demonstration test.

### Motorized Slurry Shutoff Valves

During the early development efforts on the spacecraft wet oxidation system, it became obvious that a reliable, light weight valve suitable for high pressure service in slurry systems was not available. Manually operated valves were used quite satisfactorily, but they were not available in motorized design for automatic operation. Two aerospace type ball valves were specially designed and fabricated for the NAS 1-9183 wet oxidation system by an aerospace valve manufacturer, but they proved to be a very poor design. External and internal leakage was a



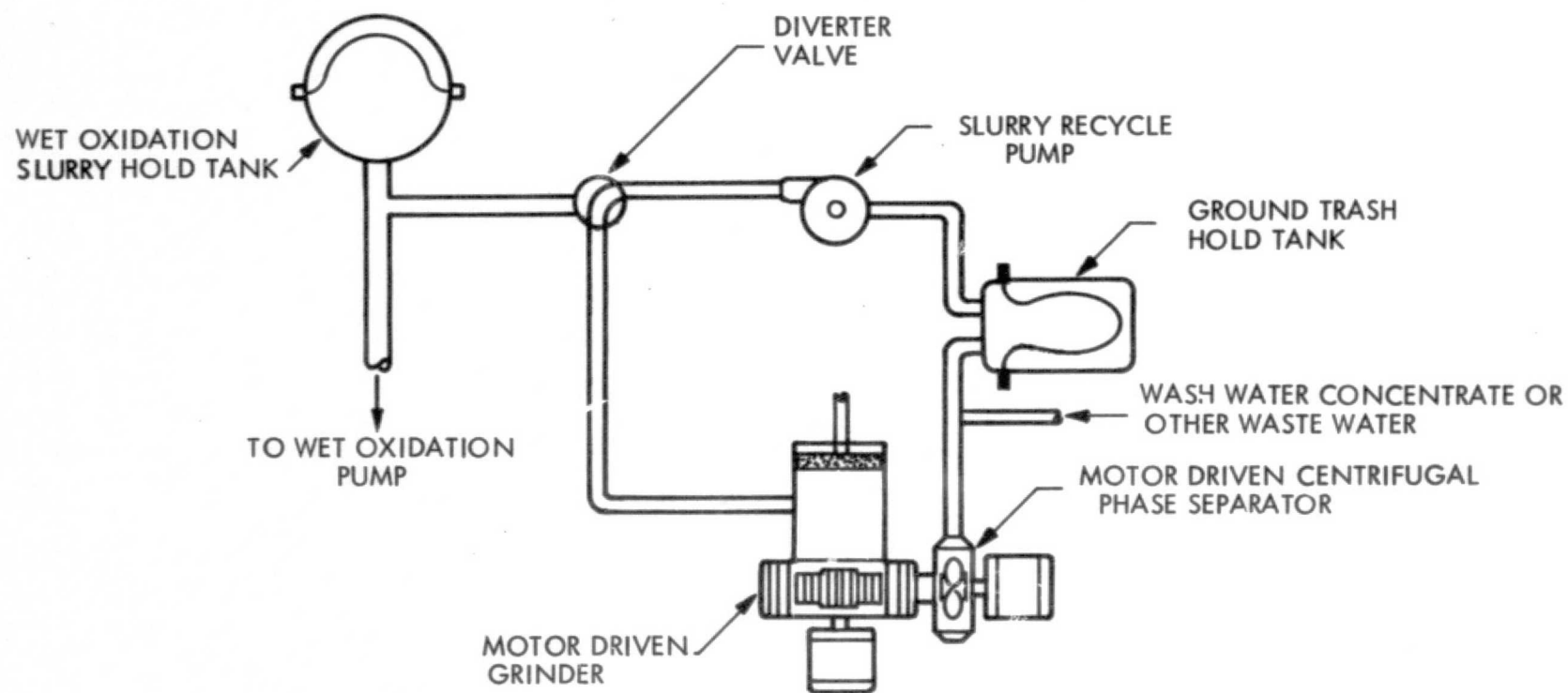


Fig. 15 Spacecraft Dry Waste Pulverizer System

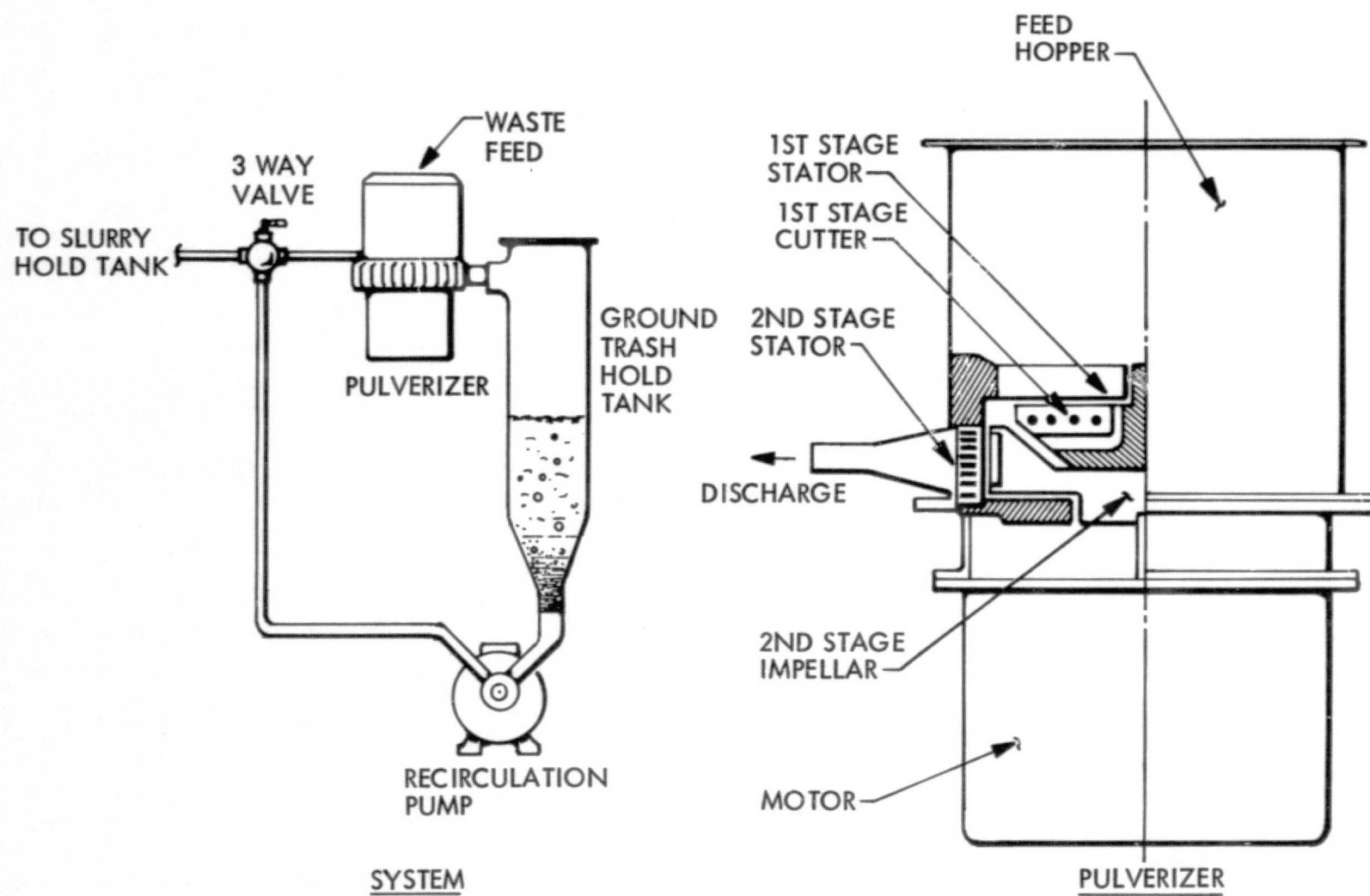


Fig. 16 Pulverizer Design Concept

reoccurring problem. It was decided that the best approach was to select a motorized actuator and attach it to the .95 cm (3/8") Whitey hand valve that had performed so well during earlier testing.

A review of available actuators resulted in the selection of the Barber Coleman actuator presented by Figure 17. It provided the required 90 degree rotation at 57.66 cm-kgs (50 in-lbs) of torque in 5 seconds in a small size with minimum weight. Another much less expensive, commercial actuator was identified as a backup. The Raymond Controls MAR 8 actuator was larger 11.15 cm x 11.15 cm x 21.1 cm (4-3/8" x 4-3/8" x 4-3/8") and weighed 1.59 kgm (3.5 lb), but was 1/10 the cost and was readily available.

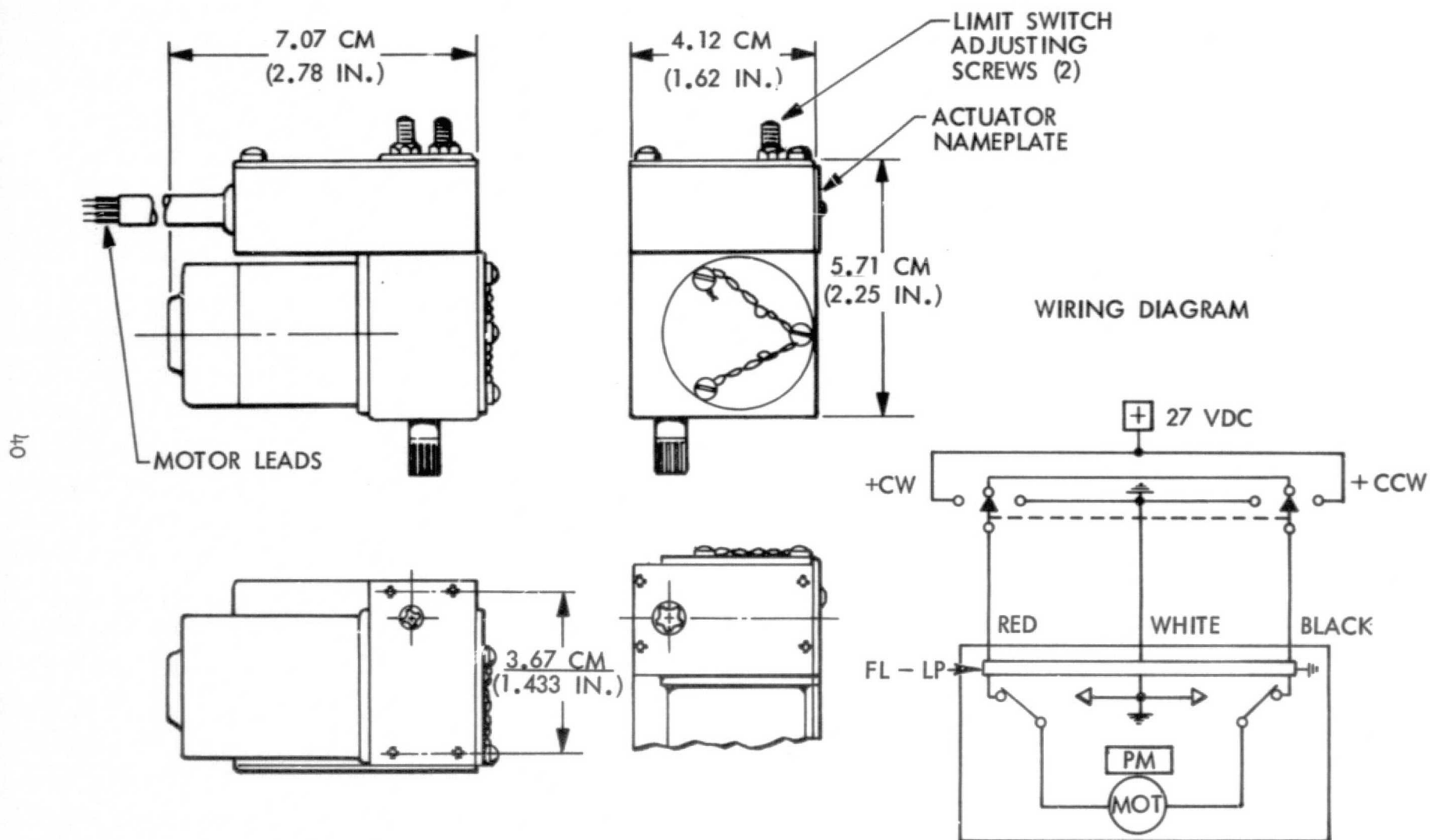


Fig. 17 Barber Coleman Rotary Actuator

## COMPONENT FINAL DESIGN

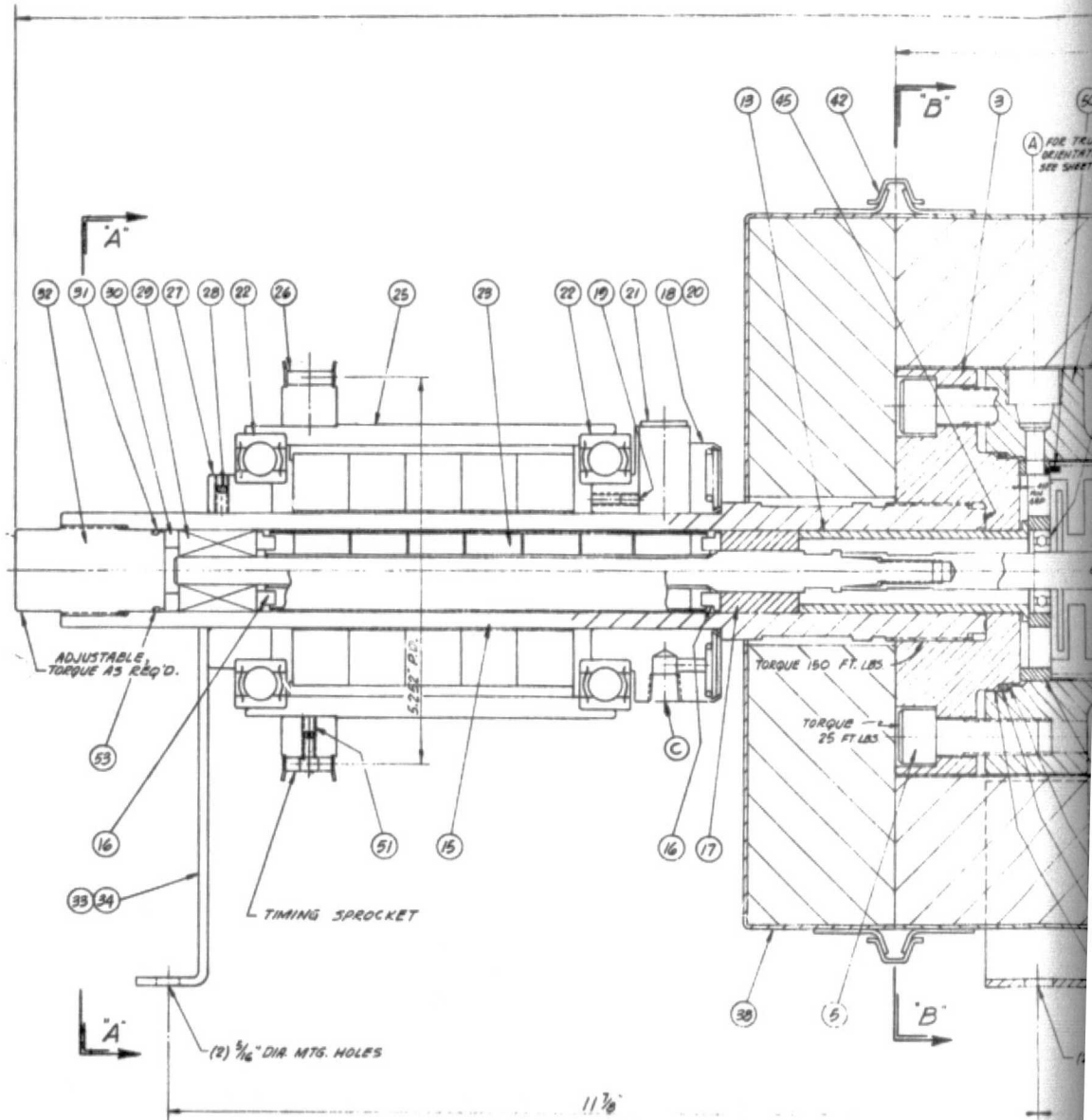
The preliminary design effort lead to final design, with emphasis on the major components. Components receiving major attention were the reactor, pulverizer, regenerative heat exchanger, catalyst introduction system, slurry pump, filters, phase separator, and motorized ball valves.

### Reactor

Figures 18 and 19 present the assembly drawings of the wet oxidation reactor that evolved from the detail design task. The reactor design was based on the stirring and drive system proven during the previous testing program, modified to provide a greater ease of disassembly and parts replacement. The previous design required that the entire reactor be disassembled to replace a bearing or end cap seal. The new design provided the ability to replace any drive tube bearing, stirring shaft bearing or end cap seal by breaking only one seal and without removing the stirring shaft or baffle assembly.

The reactor consisted of a flanged body (1), end caps (2 & 3), baffle assembly (4), stirring shaft and pin assembly (5), ball bearings (6), bearing support spider (7), drive tube housing (8), magnetic drive tube including magnets (9), drive tube radial bearings (10), drive tube thrust bearings (11), drive tube end cap (12), spacer (13), split spacer (14), external magnetic drive assembly (15), drive cooling jacket (16), external drive ball bearings (17), electrical heaters (18), insulation jacket (19), and thermocouple (20).

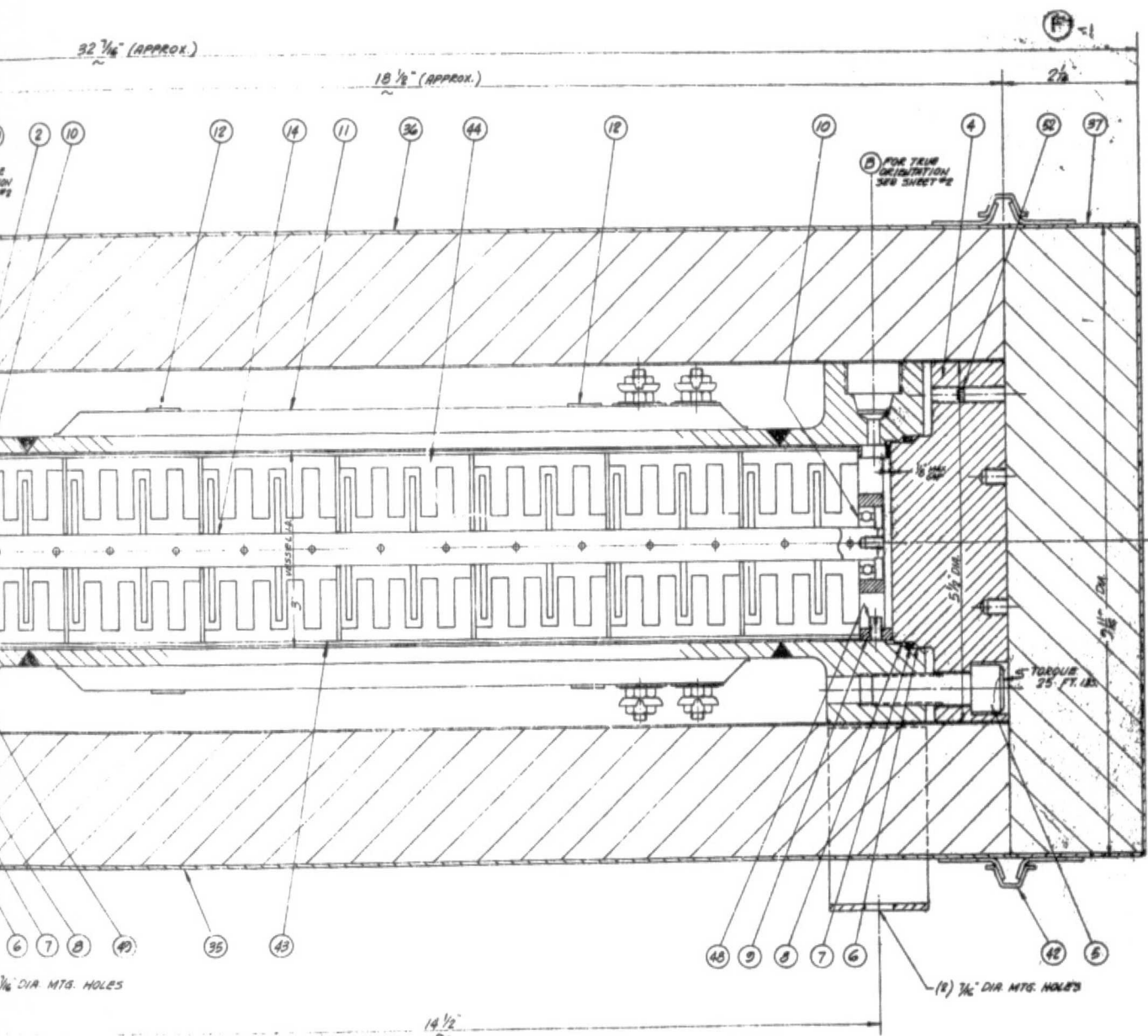
In operation, an electric motor acting through a belt drive rotated the external magnetic drive assembly which in turn caused rotation of the internal magnetic drive tube and stirring shaft. Waste materials mixed with oxygen gas entered the reactor through a 1.27 cm (1/2") tube fitting in the drive end body flange. The fluids were agitated by the combined action of the stirring rods



CONNECTION LEGEND

- (A) (1 REQ'D) 1/8" L.P. INLET
- (B) (1 REQ'D) 1/8" L.P. OUTLET
- (C) (2 REQ'D) 1/8" N.P.T. DRIVE COOLING - INLET & OUTLET
- (D) (1 REQ'D) 1/8" L.P. THERMOCOUPLE CONN.

ENCLOSURE DRAWING

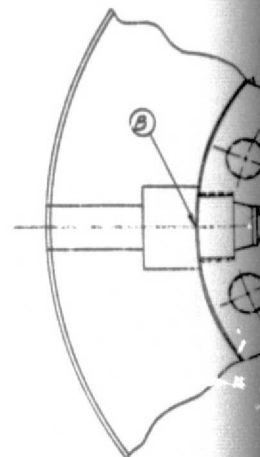


**- DESIGN SPECIFICATION -**

DESIGN PRESSURE	----- 2500 P.S.I.
DESIGN TEMPERATURE	----- 600 ° F
HYDRO-TEST PRESSURE	----- 4400 P.S.I.
CAPACITY	----- 1500 CC (APPROX.)
HEATING	----- 3 KW, 120 ° F, 1 PH.

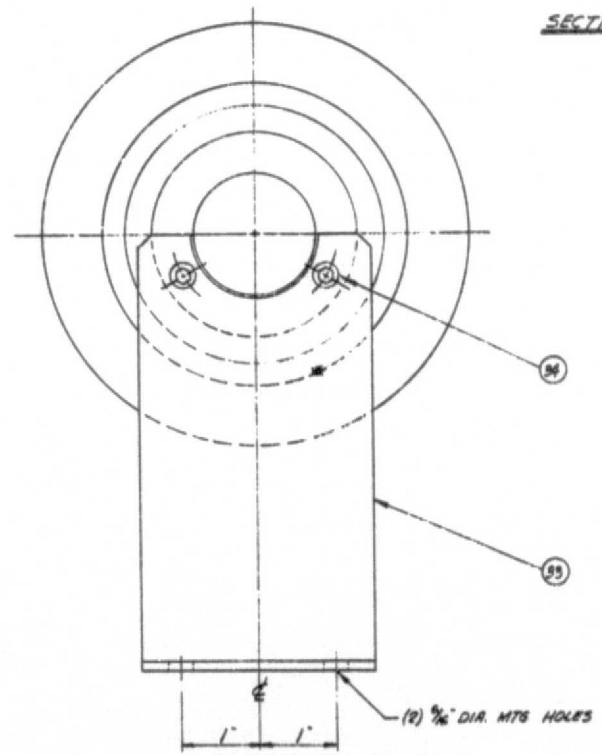
**FOULING FRAME**

Fig. 18 Reactor



SECTION THRU OUTLET  
(CONN. B')

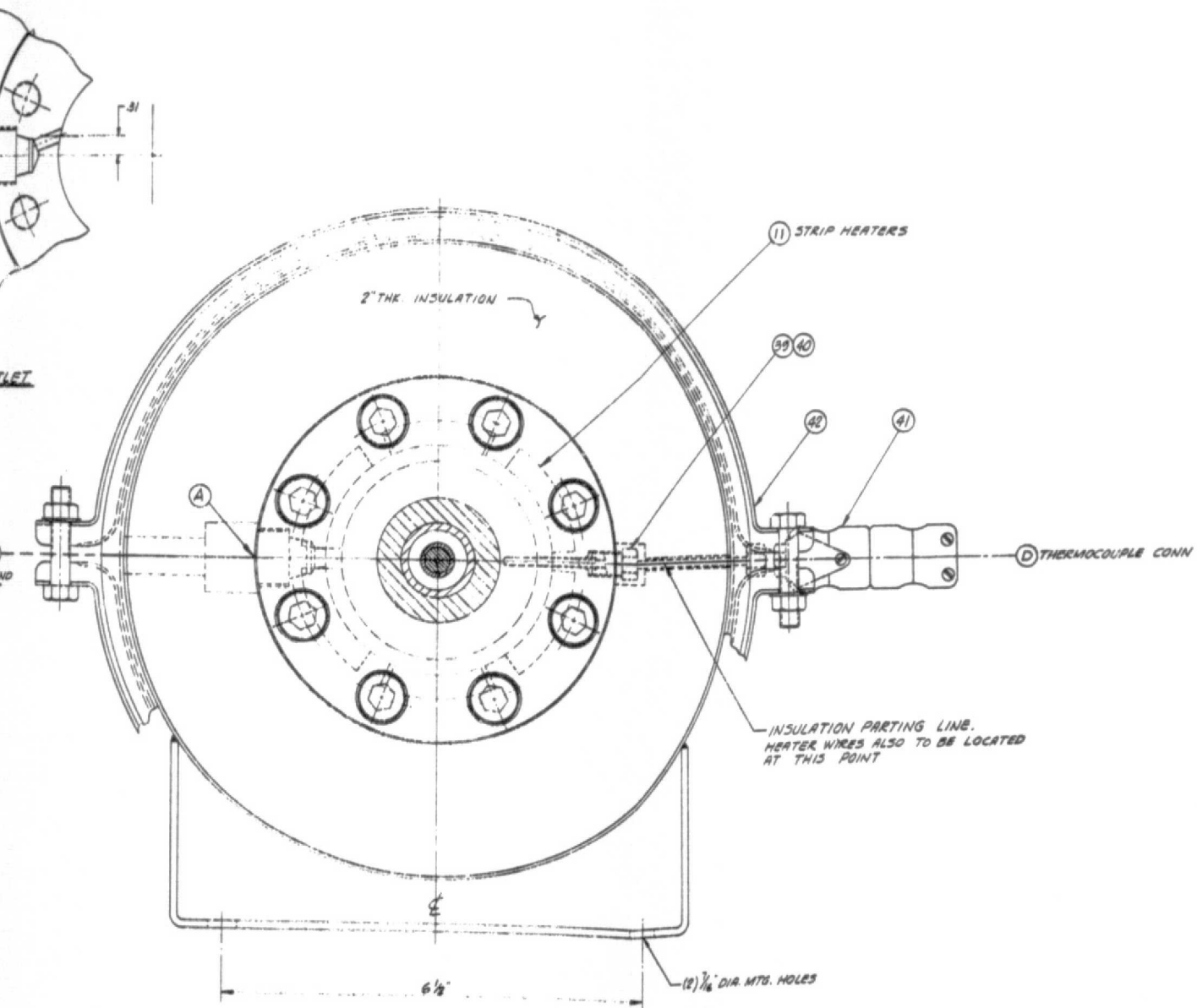
A B  
INLET AND  
OUTLET



SECTION 'A-A'

FOLDOUT FRAME





SECTION "B-B"  
ORIENTATION VIEW

FOLDOUT FRAME 2

Fig. 19 Reactor

and baffle assemblies. The incoming liquid and gas pushed the fluids in the reactor along the axis of the oxidation chamber to the outlet fitting located in the opposite body flange. The fluids passed through 0.475 cm (3/16") diameter holes in the six dams positioned across the flow path in the oxidation chamber. The dams prevented back flow of the fluid to eliminate mixing of the fluids entering the reactor with fluids leaving the reactor.

The reactor was designed to provide an internal volume of 1500 cc, giving a 30 minute residence time for a liquid flow of 1200 cc/hr. The design operating pressure and temperature were  $17.25 \text{ MN/m}^2$  (2500 psig) and  $588^\circ\text{K}$  ( $600^\circ\text{F}$ ), respectively. The reactor was designed in accordance with ASME boiler code. The drive speed was 400 RPM, as established during the previous test program. Provisions were made to vary the drive speed from 300 to 500 RPM by changing the motor pulley. The six heater elements were grouped into two sets of three to provide 3 kilowatts of power for warmup of the reactor in 45 minutes and 0.75 kw of power for sustaining temperature. The two power levels were achieved by connecting the two groups of heaters in parallel and then in series as controlled by the power control relays.

The major new design feature of the reactor assembly was the method of retaining the bearings. Previously the bearings were supported by the end caps, which meant that if the end cap was pulled to replace a seal or inspect the internal parts of the reactor the bearing had to be pulled also. The use of the spider assemblies to retain the ball bearings within the barrel of the reactor allowed the end caps to be pulled without disturbing the ball bearings and stirring shaft. The inlet and outlet ports pierced the spider assembly outer ring, which required that the spiders be positively retained. The outlet spider was set screwed to the reactor wall which acted to hold the spider and also to hold the internal parts of the reactor. The inlet spider was pinned to the reactor drive end cap to prevent it's rotation. This later proved to be a problem when the spider rotated closing off the inlet flow during the 45 day test.

All internal wetted parts including drive tube elements were fabricated from Hastelloy C-276 which was found to be resistant to the catalyzed wet oxidation environment in corrosion tests which are described in detail later in this report. The ball bearings were also made of Hastelloy C-276 to prevent electrolytic corrosion of the bearings or reactor. Considerable difficulty was encountered in locating anyone interested in fabricating the Hastelloy C-276 ball bearings on a one time basis. Industrial Tectonics Incorporated in Compton, California provided the bearings.

#### Dry Waste Pulverizer

The waste pulverizer design presented by Figure 20 was based on the preliminary design efforts and the laboratory tests conducted under contract NAS 1-9183. The pulverizer provided a rotating first stage slicer or cutter with 2 blades (1) which acted against a stationary spider with three blades (2) to reduce larger sized waste materials to smaller pieces for processing in the second stage located immediately below the first stage. The second stage impellar (3) forced the cut waste materials out through slots in the second stage cutter head (4) thereby reducing the particle size to approximately 1/16 inch or less. The pulverized wastes were forced out of the discharge tube (not shown on the drawing) by the centrifugal action of the rotating impellar acting on the waste and flush water. The first stage cutter and second stage impellar were rotated by a direct drive from a 1800 RPM 1/6 horsepower motor (5). Two sets of ball bearings (6) and (7) supported the rotating elements. Shaft seals (8) and (9) were provided to prevent leakage of liquid wastes into the motor housing. The grinder housings (10) and (11) connected the motor to the feed tube (12) as well as provided support to the bearings. The feed tube allowed waste to be loaded from the top and pushed into the first stage cutter. An automatic feeder would ultimately be used, but for testing purposes a hand held wooden piston was fabricated. A flush water connection (not shown on the drawing) provided for introduction of flush water into the first stage cutter stationary spider.

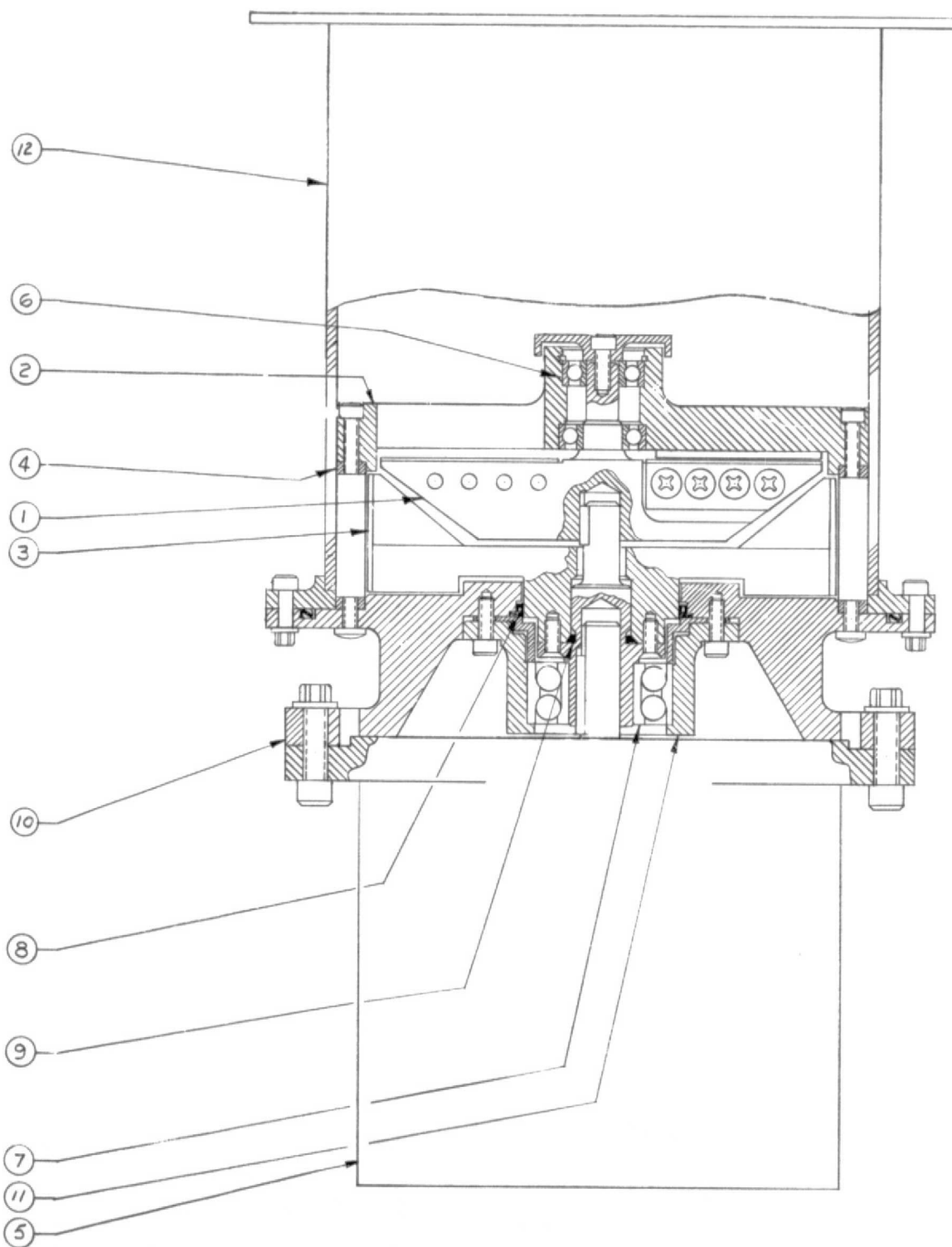


Fig. 20 Pulverizer

### Regenerative Heat Exchanger

Figure 21 presents a design drawing of the regenerative heat exchanger fabricated for use in the wet oxidation system. It represents a compromise over the smaller, lighter weight unit evolved in the preliminary design effort. 1.27 cm (1/2" O.D.) x 0.165 cm (.065") wall and 1.91 cm (3/4") O.D. x 0.125 cm (.049") wall tubes were used, because smaller tubes were not available in Hastelloy C-276. The minimum tube bend radius specified in the preliminary design was not utilized, because it would have required extensive efforts to fill the tubes with a low melting metal and preparation of special dies.

The final heat exchanger design provided a tube in tube configuration with three bends located in blocks of Johns Manville Thermal 12 insulation. An aluminum shell covered the insulation providing a box 7.43 cm (3-1/4") x 44.5 cm (18-1/8") x 94.3 cm (38-5/8").

### Catalyst Introduction System

Continued survey of vendors for a very low flow metering pump suitable for catalyst pumping finally uncovered one product, so the catalyst introduction system approach was changed during the final design effort. The pump was a Precision Control Products non metallic, diaphragm type, P/N 106 41-11. It provided an adjustable flow between 0.3 and 1.8 cc/min and was selected as the pump for catalyst injection system. The hydraulic slurry pump technique was selected to pump the slurry to high pressure, and this involves the use of bladdered tanks to hold the slurry being pumped. These are filled in about 10 min of a 6 hr cycle, so the catalyst must be injected into the slurry during the 10 min fill portion of the cycle rather than continuously as with the slide valve high pressure pump technique. The required catalyst flow rate during fill was then calculated to be 360/10 times the 0.018 cc/min calculated for the continuous flow system selected during the preliminary design. The new flowrate was 0.65 cc/min, well within the capability of the pump. Figure 22 presents a schematic of the catalyst introduction system using the diaphragm pump. The catalyst pump and solenoid valve were controlled by the timer that controlled the high pressure slurry pumping system fill and delivery valves. The solenoid valve was included in the system to prevent siphoning of the catalyst back into the hold tank when the hold tank was depressurized for filling.

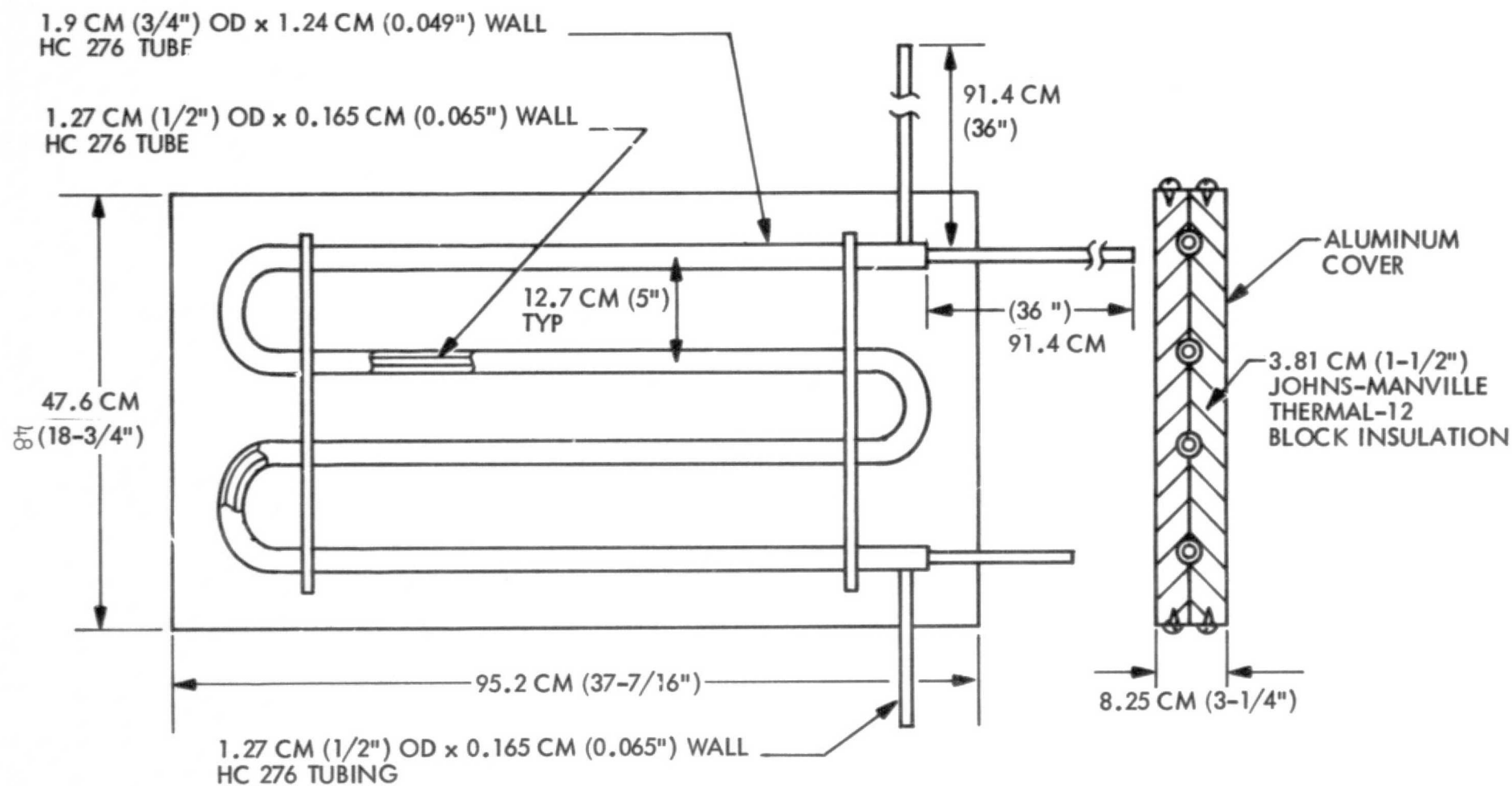


Fig. 21 Regenerative Heat Exchanger

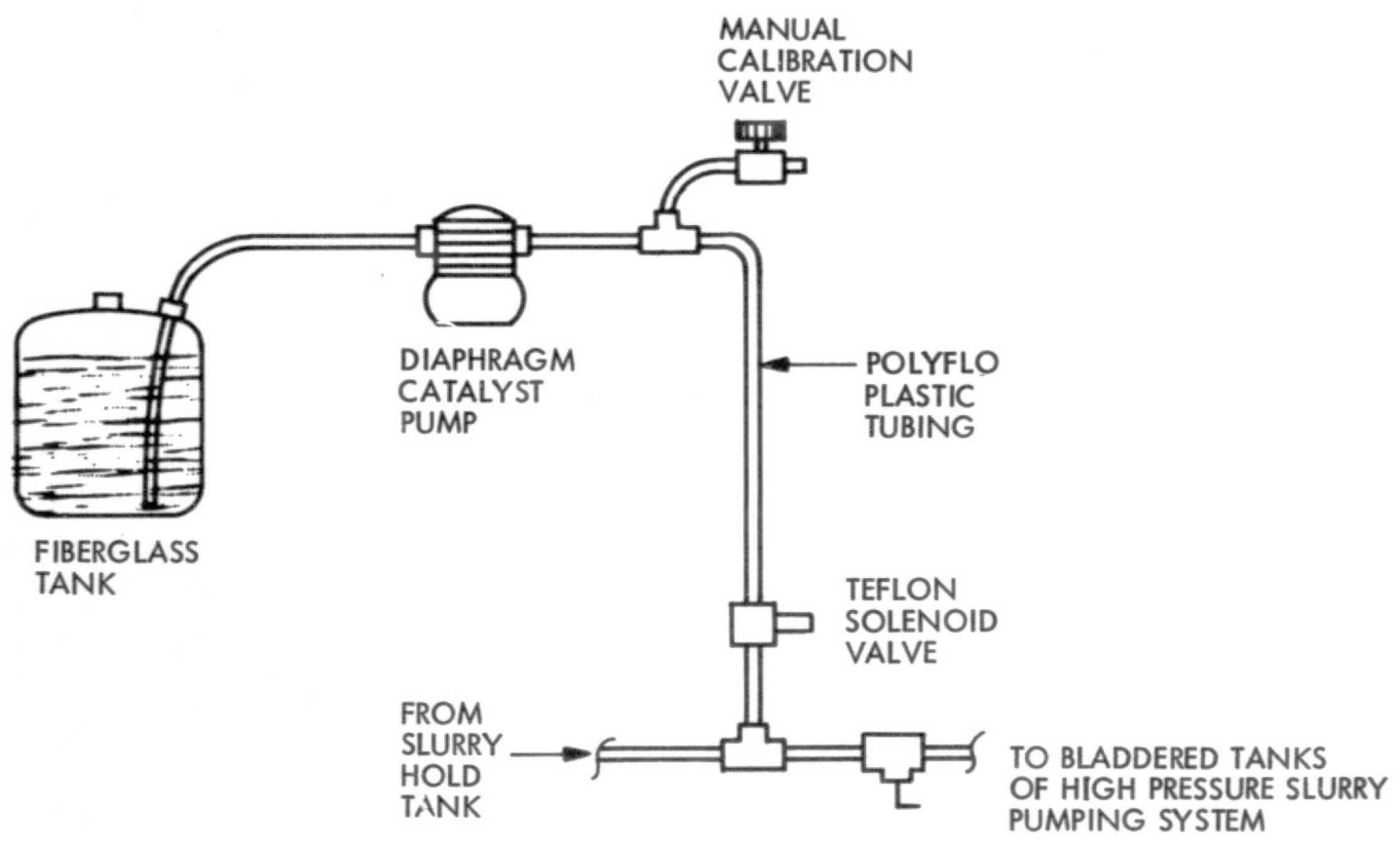


Fig. 22 Catalyst Pumping System

### Slurry Pump

The selected slurry pumping system utilized two duplex Milton Roy pumps, model 2396-89 with each head capable of delivering up to 525 cc/min at  $34.5 \text{ MN/m}^2$  (5000 psig). Figure 23 presents a photo of one of the two pumps. The four pump heads were plumbed in parallel to provide a total flow capacity of 2100 cc/min as compared to a design system slurry flowrate requirement of 1200 cc/minute.

### Motorized Ball Valves

Attempts to obtain actuators for the 0.915 cm (3/8") Whitey ball valves from Barber Coleman were not successful. They were not interested in manufacturing four of a kind on a single order, so the backup Raymond Controls MAR 8 actuator was used. Figure 24 presents a combined photo and drawing of the actuator. It provided 90 degrees of rotation in 8 seconds with an output torque of 1016.8 cm-N (90-in-lbs). The actuator was connected to the Whitey 3/8 ball valve 44S6-316 with a set screwed coupling and hat shaped bracket. The ball valves required 452 cm-N (40 in-lbs) of starting torque and 248 cm-N (22 in-lbs) of running torque. Four valve/actuator combinations were used, two for the bladdered tank/hydraulic pump, slurry pumping system and two for isolation of the hot portion of the system during emergency shutdown.

### Phase Separator

The phase separator and controller are pictured in Figure 25. They were supplied by Fluid Dynamics Corporation of Chester, California. The controller provided for manual or automatic control of the motor and solenoid valve and for adjustment of the sensor control of the solenoid valve.

### Filters

The rough and polishing filters selected for the wet oxidation system are shown in Figures 26 and 27 respectively. The rough filter is a bag type, GAF model RBX-316SS with V-5-X filter bags. The polishing filter is a Pall Trinity Micro Corporation filter MCN4463-VR-J using a MCY4463-UR filter element.



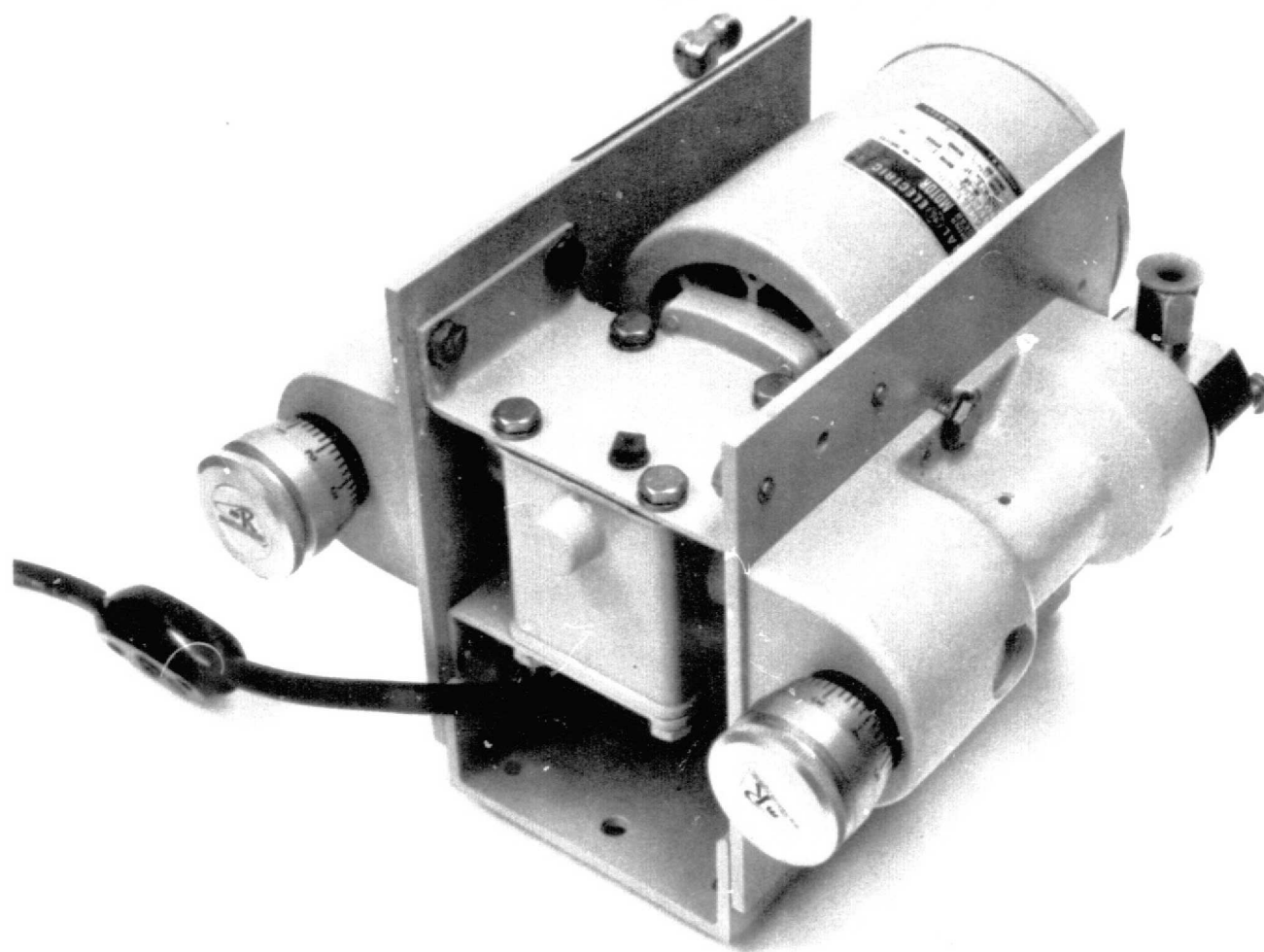


Fig. 23 Hydraulic Pump

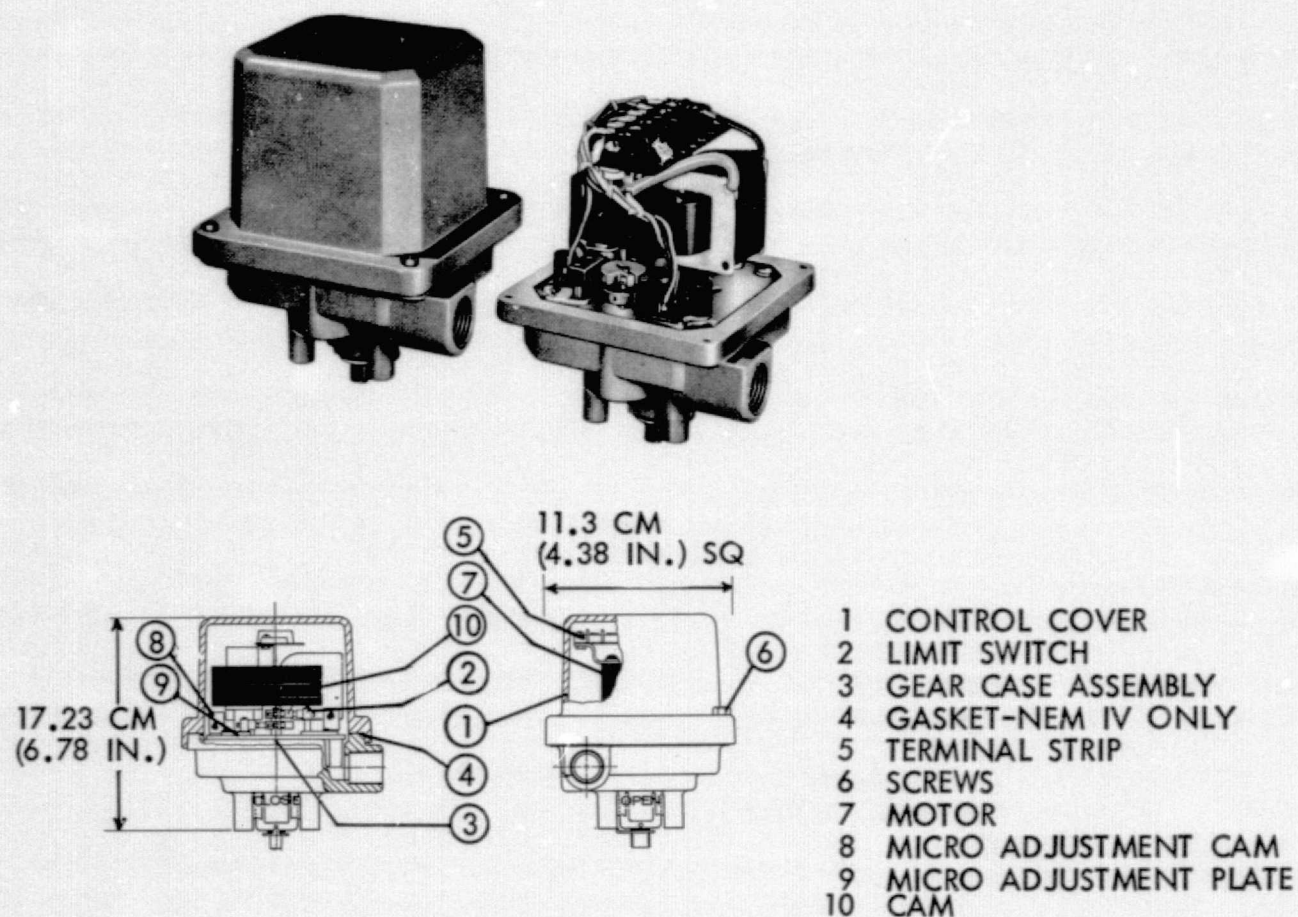


Fig. 24 Ball Valve Actuator

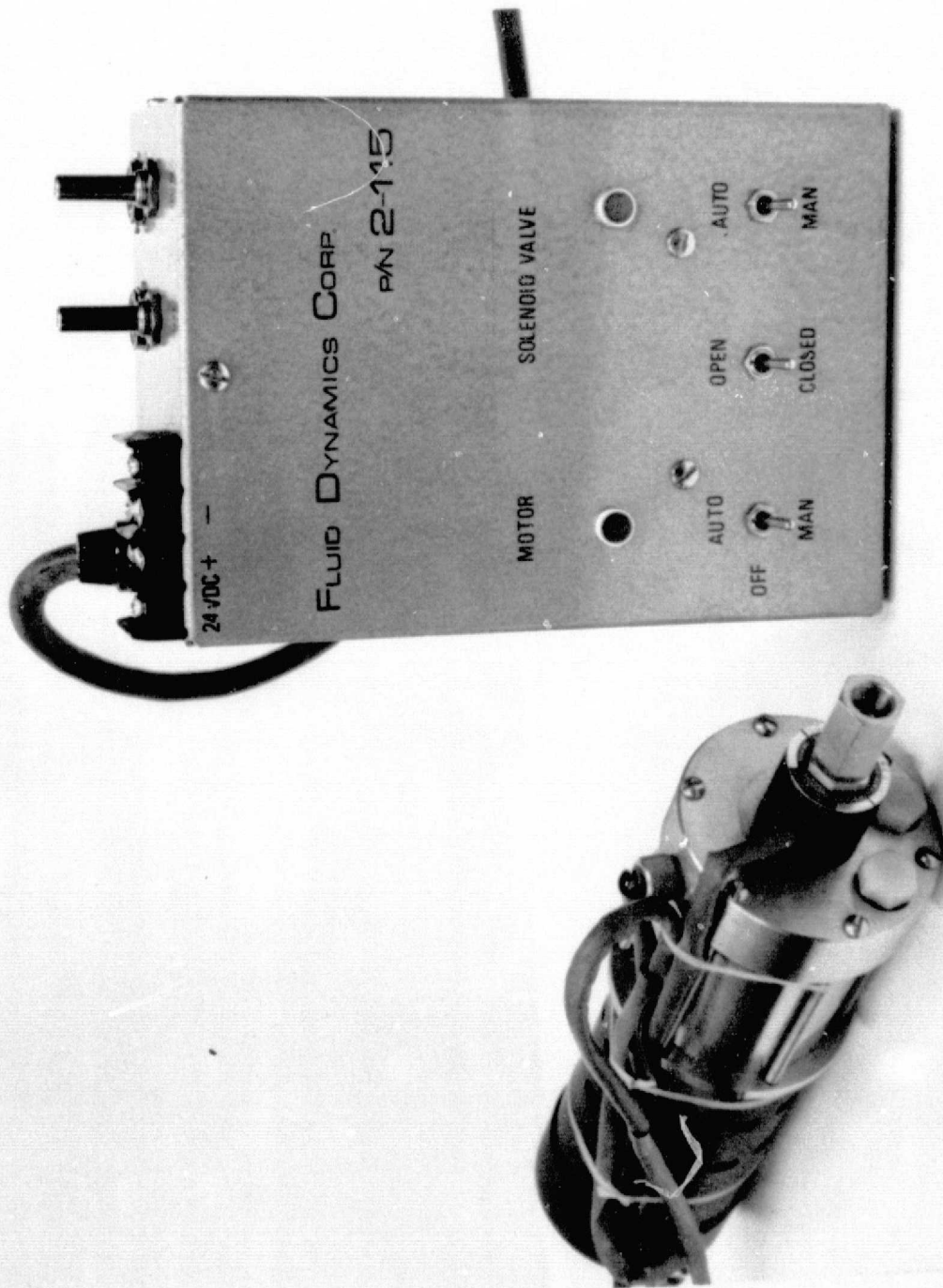


Fig. 25 Vortex Phase Separator & Controller



Fig. 26 GAF Bag Filter





Fig. 27 Pall Cartridge Filter

## SYSTEM FINAL DESIGN

Final design primarily involved the development of the system instrumentation, controls, design, failure modes and effects analysis, safety and hazards analysis, and system packaging.

### System Schematic

Figure 28 presents a schematic of the final system design. The operation of the system was as follows. The pulverizer slurry tank (26) was partially filled with water, and trash was loaded into the pulverizer feed chamber. The pulverizer (24) and pulverizer recirculation pump (31) were started. The three-way valve (33) was positioned to recirculate the water/ground trash mixture back into the pulverizer to help in pulverizing additional trash and to further reduce trash particle size. After pulverizing a full load of trash in accordance with the trash model, the three way valve (33) was positioned to allow the pump (31) to empty the pulverized trash into the main slurry hold tank (58). When the pulverizer hold tank was empty, the pulverizer and pump were de-energized and the three way valve was returned to the recirculation mode. Feces, urine and flush water in accordance with the waste model were mixed externally to the system and poured into the main slurry hold tank (58) through a fitting on the top of the tank. The tank was then pressurized to 20 psig to allow delivery of the slurry to the wet oxidation slurry pumping system through manual shutoff valve (9). During testing the slurry hold tank was filled once each day. The slurry was forced into two bladdered tanks (29) when the motorized shutoff valve (10) was opened by the timer (45) for a fill cycle. Simultaneously, the bladder pushed water on the back side, out of the slurry tanks (29) through the open solenoid valve (20) into the water reservoir (35). After a time sufficient to insure tank filling, the timer (45), closed the fill valve (10) and solenoid valve (20), allowing pump (30) to pressurize the bladdered tanks (29) to the relief valve (21) setting of 2500 psig. The timer then opened motorized valve (11) to allow delivery of the slurry to the reactor system.

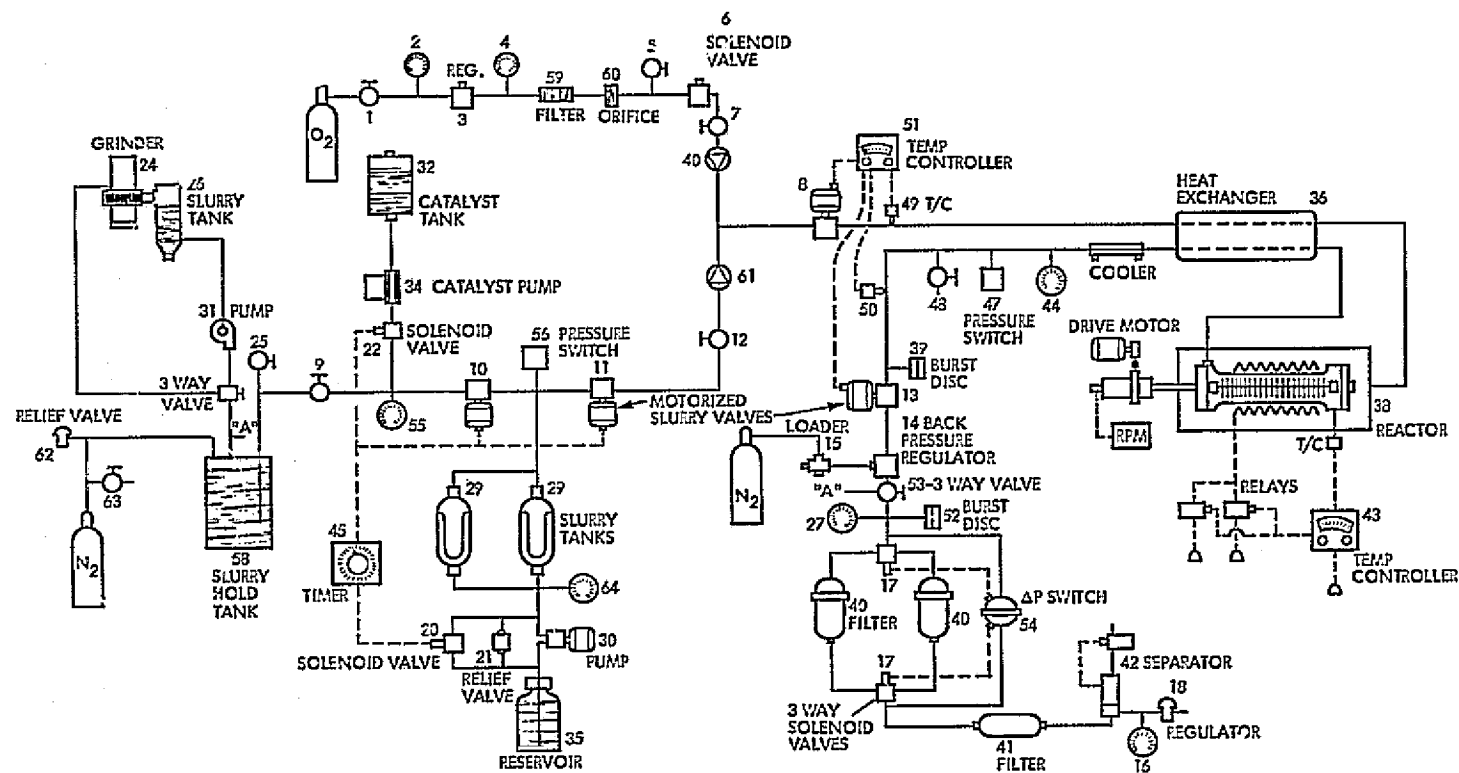


Fig. 28 Wet Oxidation System Schematic

The feed rate of slurry was established by adjusting the stroke of the pistons on the Milton Roy slurry pumps (30). Six hours later (time required to empty the slurry tanks at design slurry feed rate) the timer closed valve (11), and opened valves (10) and (20) for another fill cycle. Catalyst from storage tank (32) was pumped by the diaphragm pump (34) through solenoid valve (22) into the slurry feed line upstream of motorized valve (10). The catalyst pump and solenoid valve were energized and de-energized by the timer (45) at the same time that valve (10) was opened and closed.

Oxygen from a high pressure (3000 psi) storage bottle was passed through shutoff valve (1) and reduced in pressure to approximately 2400 psig by oxygen regulator (3). The oxygen was then passed through a 5 micron filter (59) prior to flowing through a calibrated sintered metal orifice (60). The pressure downstream of the orifice was controlled by the reactor back pressure controller so that oxygen flowrate was dependent upon the orifice upstream pressure as determined by regulator (3). Oxygen solenoid valve (6) was provided to allow oxygen shutoff during system automatic shutdown. Check valves (40) and (61) were provided in the oxygen and slurry feed lines to prevent backflow in case of a serious leak in the upstream plumbing. The oxygen and slurry were mixed before passing through a safety isolation valve (8) and temperature sensor (49) that shut valve (8) if the temperature exceeded  $322^{\circ}\text{K}$  ( $120^{\circ}\text{F}$ ) as measured by sensor (49). High temperatures at this point were used as an indication of back flow from the hot downstream portion of the system. The combined sewage/oxygen mixture was passed through the tubular regenerative heat exchanger (36) where it was heated to near the reactor temperature of  $560^{\circ}\text{K}$  ( $550^{\circ}\text{F}$ ). The hot mixture then entered the stirred reactor (38) where it was agitated for  $1/2$  hr at  $560^{\circ}\text{K}$  ( $550^{\circ}\text{F}$ ) at a total pressure of  $15.2 \text{ MN/m}^2$  (2200 psig) in order to oxidize the waste materials.

The internal chamber of the reactor vessel was divided into seven chambers to provide as closely as possible a plugged flow reactor to prevent input wastes from mixing with outlet fluids. The stirring shaft with stirring pins attached passed through the seven chambers. Baffles were provided between each stirring pin to aid in agitation of the waste/oxygen mixture. A temperature controller,



relays, and heaters were provided to heat the reactor to  $560^{\circ}\text{K}$  ( $550^{\circ}\text{F}$ ) initially and to maintain the temperature during operation of the system. An electric motor, drive belt, and external magnet assembly provided for rotation of the internal stirring shaft without the necessity of a rotating shaft seal.

The hot reactor effluent liquid and gases were passed through the outlet pass of the regenerative heat exchanger where they were cooled by the incoming sewage/oxygen mixture. A water cooled tubular cooler was used to further cool the effluent fluids. A pressure switch (47) for safety shutdown, a bleed valve (48), pressure gage (44), temperature sensor (50), burst disc (39) for safety pressure release, and emergency shutdown valve (13) were provided downstream of the cooler. The temperature sensor (50) acting through the temperature controller (51) shut the isolation valves (8) and (13) if the outlet temperature exceeded  $322^{\circ}\text{K}$  ( $120^{\circ}\text{F}$ ). Temperatures at or above  $322^{\circ}\text{K}$  ( $120^{\circ}\text{F}$ ) at this point indicated a failure of the back pressure regulator or the presence of a bad leak in the outlet line. The back pressure regulator (14) maintained the reactor system at a nominal pressure of  $15.2 \text{ MN/m}^2$  (2200 psig). A pressure loader (15) and high pressure nitrogen source provided the means for loading the gas loaded regulator (14). Three way valve (53) allowed recirculation of the effluent fluids back to the slurry hold tank during startup of the system. A low pressure burst disc set at  $690 \text{ kN/m}^2$  (100 psig) protected the filters (40) in case the back pressure regulator failed to hold system pressure. The filters removed the fine ash generated by the oxidation process. Two-three way solenoid valves (17) and a  $\Delta P$  switch (54) provided for switching from one filter to the other. If  $\Delta P$  rose to  $276 \text{ kN/m}^2$  (40 psig), the solenoid valve positions were automatically changed to bring a fresh filter on line and signal for a filter bag replacement. Polishing filter (41) was located downstream of the coarse bag filters (40). The centrifugal phase separator (42) received liquid and gas from the polishing filter. Liquid from the separator was vented through a back pressure regulator (18) which maintained a pressure of  $138 \text{ kN/m}^2$  (20 psig) on the separator to simulate the pressure in an effluent hold tank. Gas from the separator was vented to the room.

## System Instrumentation & Controls

Table 4 presents the controls for the normal operation of the wet oxidation system. Basically the control approach was very simple. The oxygen and slurry were continually forced into the reactor at constant flows and the back pressure regulator vented the liquid and gas effluents to maintain reactor pressure. The oxygen regulator (3) maintained a constant pressure on the upstream side of the flow orifice and the downstream pressure was maintained by the system back pressure regulator (14), thereby producing a constant oxygen flow within the limits of pressure control by the two regulators. The slurry flow was controlled by the positive displacement piston hydraulic pumps (30) which forced water on the back side of the bladdered tanks (29) at a constant rate. The timer (45) controlled the action of the motorized slurry valves (10 and 11), pump bypass solenoid valve (20), catalyst pump (34), and catalyst solenoid valve (22) to fill the bladdered tanks every six hours with a catalyzed sewage/trash mixture. The reactor thermocouple and temperature controller (43) activated a series of relays to connect two sets of reactor heaters in parallel, providing 3 kilowatts of power for warmup, and in series for sustaining reactor temperature.

Additional controls for normal operation included switching of the filter solenoid valves (17) by the filter  $\Delta P$  switch set at  $276 \text{ kN/m}^2$  (40 psi) and control of the phase separator gas vent solenoid valve by the vortex pickup and controller. Back pressure regulator (18) controlled the discharge of effluent liquid to simulate the pressure of an effluent water tank.

Emergency controls as shown by Table 5, included a low pressure contact on pressure switch (56) to prevent motorized valve (10) from being opened for a bladdered tank (29) refill unless the pressure on the tanks was less than  $690 \text{ kN/m}^2$  (100 psi). A high pressure contact of the same pressure switch (56) was used to prevent motorized valve (11) from being opened unless the tank pressure was  $14.5 \text{ MN/m}^2$  (2100 psig) or greater to prevent backflow from the hot reactor. Automatic system shutdown or alarm was provided by signals from any of the fail safe shutdown sensors shown by Table 5. These sensor output signals were used to close the isolation valves

Table 4 System Controls\*

(Normal Operation)

<u>Controller</u>	<u>Controlled Element</u>	<u>Controlled Condition</u>
Oxygen Regulator (3)	-	o Maintained constant pressure on orifice
Timer (45)	{ Catalyst Pump (34) Catalyst Solenoid (22) Slurry Inlet Valve (10) Slurry Outlet Valve (11) Hydraulic Pump Bypass (20)	o Cycled every six hours for slurry tank refill
Reactor Temperature Controller (43)	Reactor Heaters	{ o Two sets in parallel for reactor warmup o Two sets in series for sustaining temperature
Back Pressure Regulator (14)	-	o Maintained constant reactor pressure
Filter $\Delta P$ Switch (54)	Filter Solenoid Valves (17)	o Switched filters on $276 \text{ kN/m}^2$ (40 psi) $\Delta P$
Phase Separator Impellar RPM	Phase Separator Gas Valve	o Open on low RPM
Back Pressure Regulator (18)	-	o Maintained constant back pressure on effluent water

\*numbers refer to parts in schematic, Figure 28.

Table 5 Emergency Controls\*

<u>Controller</u>	<u>Controlled Element</u>	<u>Controlled Condition</u>
Pressure Switch (56) (Low Setting $13.8 \text{ MN/m}^2$ , 2000 psig)	Slurry Inlet Valve (10)	o Cannot open valve unless tank (29) pressure is less than 100 psi
Pressure Switch (56) (High Setting $16.56 \text{ MN/m}^2$ , 2400 psig)	Slurry Outlet Valve (11)	o Cannot open valve unless tank (29) pressure is greater than 2100 psi
Temp Controller (51) (High Setting, $322^\circ\text{K}$ , $120^\circ\text{F}$ )	Isolation Valves (8 & 13)	<div> o Day mode - close valves 8 &amp; 13 and sound alarm o Night mode - close valves 8 &amp; 13 and turn off main power </div>
Temp Controller (43) (High Setting, $588^\circ\text{K}$ , $600^\circ\text{F}$ )		
Pressure Switch (47) (High Setting, $16.56 \text{ MN/m}^2$ , 2400 psig)		
Pressure Switch (47) (Low Setting, $13.8 \text{ MN/m}^2$ , 2000 psig)		
Filter $\Delta P$ Switch (54) (High Setting, $414 \text{ N/m}^2$ , 60 psi)		

\*numbers refer to parts in schematic, Figure 28

(8 and 13) and shutoff all power if in the "night" control mode and ring an alarm if in the "day" control mode. When an operator was on duty during the day it was considered better not to shutdown completely, but to give him a signal that something was wrong and let him fix it and get the system back on line with a minimum interruption. For night operation or when the operator was not on duty, it was considered to be better to shutdown completely. Power off, with the isolation valves closed, was a safe shutdown condition. Isolation valve power was separated from the main power source, so that isolation valve closure and de-energizing of the main power could be accomplished simultaneously.

Table 6 presents the instrumentation provided for monitoring system operation and for adjustment of the controls.

#### Electrical System

The electrical system schematic is presented by Figure 29. All controls and electrical equipment used 110V, 60Hz, 1 $\phi$  ac power, except for the phase separator which also required 28V dc and the hydraulic pumps which required 208V, 60Hz, 1 $\phi$  ac. All components were wired for automatic control with manual override. The hydraulic pumps, phase separator, reactor drive motor, filters solenoid valves, and fail safe shutdown controls were wired to operate any time that the main power switch was closed. The catalyst pump and solenoid valve, the slurry pump components, and the oxygen solenoid valve were controlled by the timer. The reactor heaters were controlled by the reactor temperature controller. The fail safe shutdown controls turned off all power and closed the isolation valves if the night mode was selected and rang an alarm and closed the isolation valves if day control was selected.

#### System Packaging

The system was packaged and arranged in four modules for the test program. Figure 30 is a photo of the test setup. The oxygen and nitrogen supply tanks and slurry hold tank formed one module. The pulverizer subsystem consisting of pulverizer, hold tank, recirculation pump and three way valve constituted the second module. These two modules were not as sophisticated with respect to

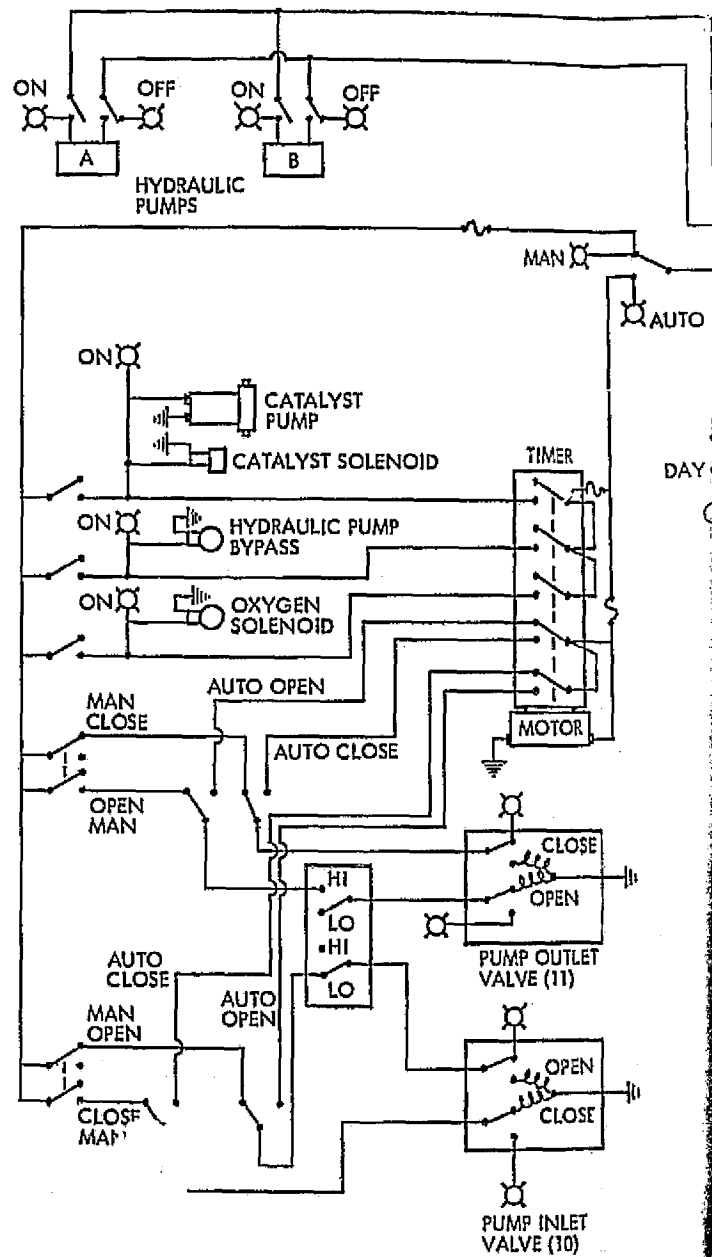
Table 6 System Instrumentation

Direct Reading

Oxygen Supply Pressure (2)  
Regulated Oxygen Pressure (4)  
Slurry Hold Tank Pressure (55)  
Slurry Tank Fill Pressure (55)  
Bladdered Tank Pressure (64)  
Slurry Hold Tank Level (visual)  
Hydraulic Reservoir Level (visual)  
Timer Position (45)  
Motorized Valve Position Indicators (10), (11), (8), (13)  
Reactor Drive RPM  
Reactor Temperature  
Reactor Heater Current  
Reactor Heater Voltage  
Reactor Pressure (44)  
Filter Inlet Pressure (27)  
Effluent Water Pressure (16)

Panel Light Indicators

Isolation Valve Position (8)(13)  
Filter Solenoid Valve Power On or Off (17)  
Reactor Heater Power (High or Low Heat Indication)  
Catalyst Pump Power On or Off (34)  
Hydraulic Pump "A" Power On or Off (30)  
Hydraulic Pump "B" Power On or Off (30)  
Hydraulic Pump Bypass Solenoid Power On or Off (20)  
Oxygen Solenoid Valve Power On or Off (6)  
Slurry Inlet Valve Position (10)  
Slurry Outlet Valve Position (11)



SLURRY, OXYGEN, & CATALYST SUPPLY CONTROLS

**FOLDOUT FRAME**

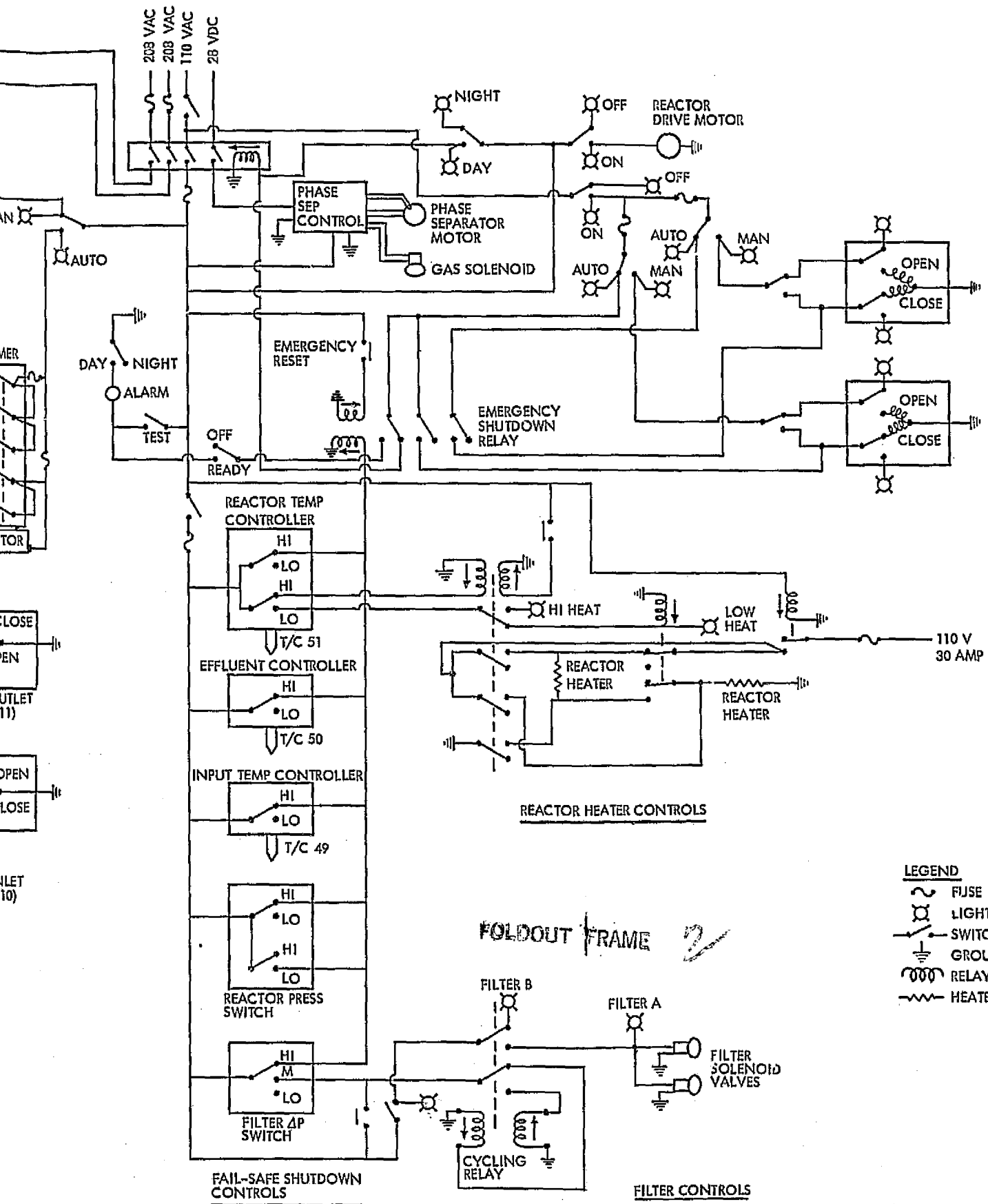


Fig. 29 Electrical System Schematic



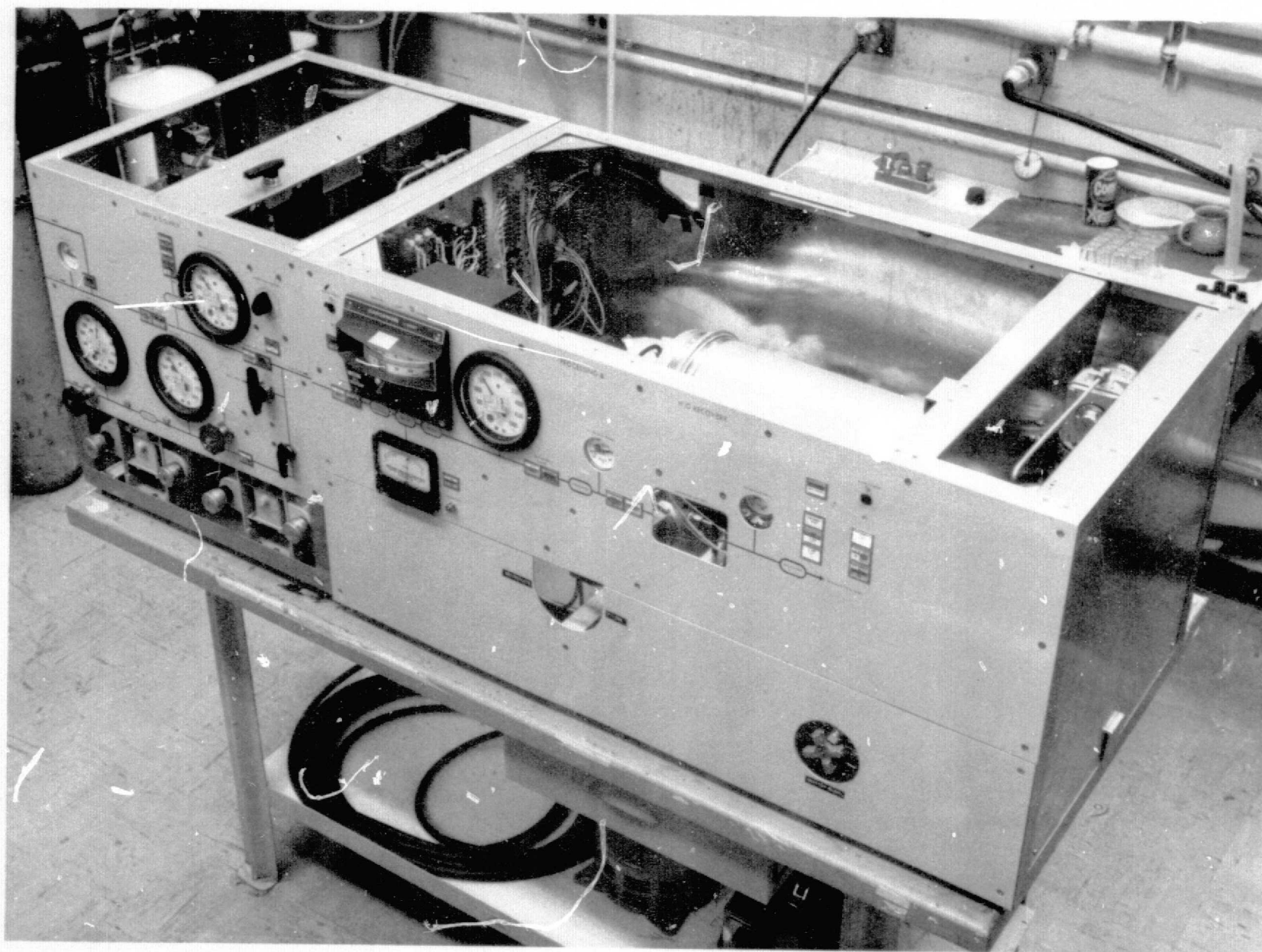


Fig. 30 Demonstration System Test Modules

hardware design or arrangement as the remaining two and were considered to be more laboratory or experimental. The remaining two modules, representing the heart of the system, were packaged to simulate a spacecraft installation. The smaller module on the left of Figure 30 housed the slurry, oxygen and catalyst supply subsystems. The larger module on the right contained the isolation valves, heat exchanger, reactor, cooler, back pressure regulator, filters, phase separator, effluent regulators, and associated hardware. All displays and controls associated with the components in a given module were located on the front of that module. Figures 31 and 32 are sketches of the equipment layouts for the slurry, oxygen, and catalyst supply module and the processing and water recovery module.

Table 7 presents weight, volume, and power estimates made for the system as shown in Figure 30 and for a flight type system considering that most of the equipment used in the demonstration test system is not designed for flight application and considerable weight, volume and power savings could be realized by redesign. Weight, volume and power estimates are 203 kgm (447.1 lb)  $0.735 \text{ m}^3$ , (26 ft.<sup>3</sup>) and 520 watts for the demonstration system and 93.34 kgm, (205.6 lb),  $0.4888 \text{ m}^3$  (17.3 ft.<sup>3</sup>) and 285 watts for a flight system based on a slurry processing rate of 27.83 kgm, (61.3 lb) per day.

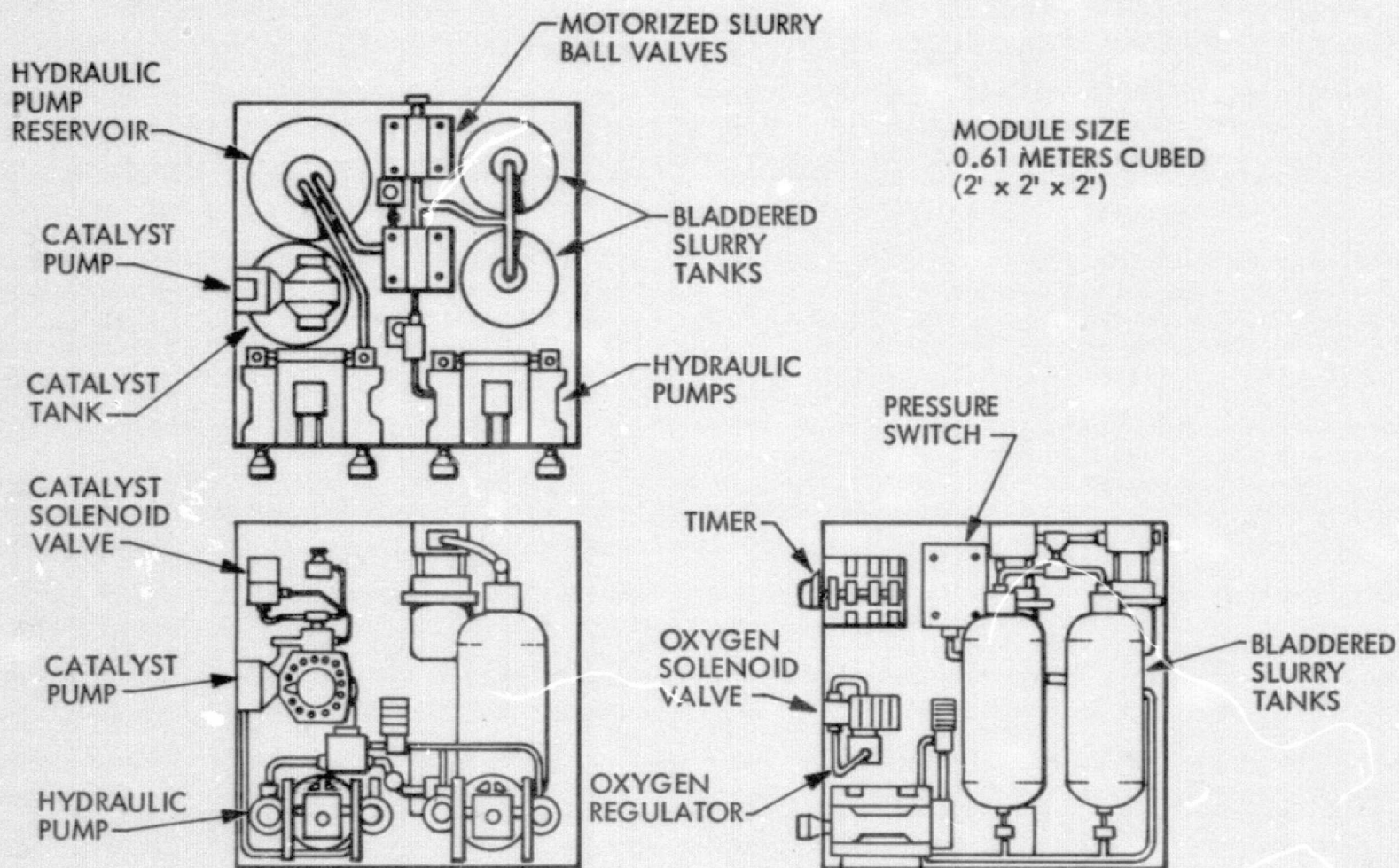


Fig. 31 Oxygen, Catalyst & Slurry Supply Module





Fig. 32 Processing and Product Recovery Module

Table 7

Weight, Power, and Volume Statement  
For NASA Wet Oxidation Waste Management System

Description	Current Weight kgm(lb)	Potential Weight kgm(lb)	Steady State Power	
			Current (watt)	Potential (watt)
Hydraulic Reservoir	1.36 (3.0)	0 (0)		
Hydraulic Pumps (2)	19.98 (44.0)	9.1 (20)	40	(0)
Hydraulic Relief Valves (2)	2.27 (5.0)	0 (0)		
Hydraulic Bypass Solenoid Valve	1.59 (3.5)	0 (0)		
Hydraulic Pressure Switch	1.14 (2.5)	0.23 (0.5)		
Hydraulic Pressure Gage	0.82 (1.8)	0.45 (1.0)		
Oxygen Shut-off Valves (2)	0.68 (1.5)	0.68 (1.5)		
Oxygen Aux. Inlet Valve	0.36 (0.8)	0 (0)		
Oxygen Pressure Gage (2)	1.63 (3.6)	0.91 (2.0)		
Oxygen Pressure Regulator	1.82 (4.0)	0.68 (1.5)		
Oxygen Filter	0.18 (0.4)	0.18 (0.4)		
Oxygen Restrictor (2)	0.14 (0.3)	0.05 (0.1)		
Oxygen Bleed Valve	0.32 (0.7)	0.32 (0.7)		
Oxygen Solenoid Valve	1.59 (3.5)	0 (0)	44	(0)
Oxygen Check Valve	0.14 (0.3)	0.14 (0.33)		
Catalyst Tank	2.32 (5.1)	2.32 (5.1)		
Catalyst Pump	3.63 (8.0)	0.91 (2)		
Catalyst Solenoid Valve	0.45 (1.0)	0.45 (1.0)		
Slurry Accumulator Bladder Tanks (2)	10.4 (23.0)	0 (0)		
Slurry Motor Actuated Valves (2)	4.54 (10.0)	0 (0)		
Slurry Hand Actuated Valves (2)	1.09 (2.4)	1.09 (2.4)		
Slurry Check Valve	0.45 (1.0)	0.23 (0.5)		
Slurry Pressure Gage	0.22 (0.5)	0.23 (0.5)		
Terminal Board	0.22 (0.5)	0.23 (0.5)		
Wiring (Total Module)	1.0 (2.2)	0.68 (1.5)	20	(14)
Timers (2)	1.36 (3.0)	0.23 (0.5)	5	(1)
Pushbutton Switch/Lites (10)	0.82 (1.8)	0.23 (0.5)	1	(1)
Plumbing (Total Module incl. Fittings)	1.82 (4.0)	1.36 (3.0)		
Structure	9.08 (20.0)	4.54 (10)		
Total Dry Weight Supply Module	71.46 (157.4)	25.2 (55.5)	110	(16)
Hydraulic Fluid	7.58 (16.7)	0 (0)		
Catalyst Solution	0.91 (2.0)	0.91 (2.0)		
Slurry	7.58 (16.7)	0.45 (1.0)		
Total Liquids	16.07 (35.4)	1.36 (3.0)		
Total Weight Supply Module (Wet)	87.53 (192.8)	26.56 (58.5)	110	(16)
Total Supply Module Volume	0.3m <sup>3</sup> (8.67ft <sup>3</sup> )	0.21m <sup>3</sup> (6.1ft <sup>3</sup> )		

Table 7 (continued)

Description	Current	Potential	Steady State Power	
	Weight kgm(lb)	Weight kgm(lb)	Current (watt)	Potential (watt)
Reactor (Including Drive Motor, Insulation, Fan, Belt, RPM Meter, Tach. Adjust)	40.86 (90.0)	29.5 (65)	265	(200)
Reactor Motor Actuated Valves (2)	4.54 (10.0)	2.27 (5)		
Reactor Regenerative Heat Exchanger	22.7 (50.0)	11.4 (25)		
Reactor Back Pressure Regulator	1.32 (2.9)	0.91 (2)		
Reactor Bleed Valve	0.32 (2.9)	0.32 (0.7)		
Reactor 3-Way Recycle Valve	0.22 (0.5)	0.22 (0.5)		
Reactor Pressure Switch	1.14 (2.5)	0.22 (0.5)		
Reactor Pressure Gage	0.82 (1.8)	0.45 (1.0)		
Reactor High Pressure Burst Disc	0.45 (1.0)	0.45 (1.0)		
Reactor Regen. Ht. Exchgr. Thermocouples (2)	0.09 (0.2)	0.09 (0.2)		
Reactor Regen. Ht. Exchgr. Temp. Alarm Controller (2)	1.36 (3.0)	0.68 (1.5)	10	(5)
Reactor Temp. Controller	3.86 (8.5)	0.45 (1.0)	15	(10)
Cooler	0.77 (1.7)	0 (0)		
Low Pressure Burst Disc	0.91 (2.0)	0.22 (0.5)		
Filter Primary (2)	3.63 (8.0)	1.82 (4.0)		
Filter Secondary	1.14 (2.5)	0.45 (1.0)		
Filter Solenoid Valves	3.18 (7.0)	0.91 (2)	44	(0)
Filter Pressure Gage	0.22 (0.5)	0.22 (0.5)		
Filter Pressure Switch	1.14 (2.5)	0.22 (0.5)		
Phase Separator	2.32 (5.1)	1.82 (4)	27	(20)
Phase Separator Control	0.5 (1.1)	0.22 (0.5)	2	(2)
Phase Separator Pressure Gage	0.22 (0.5)	0.22 (0.5)		
Phase Separator Liquid Back Press. Relief Valve	0.14 (0.3)	0.14 (0.3)		
Pushbutton Switch/Lites (15)	1.23 (2.7)	0.32 (0.7)	2	(2)
Circuit Breakers (2)	0.18 (0.4)	0.18 (0.4)		
Alarm	0.45 (1.0)	0.22 (0.5)		
Relays (6)	0.54 (1.2)	0.22 (0.5)	5	(2)
Terminal Board	1.04 (2.3)	0.91 (2)		
Wiring (Total Module)	2.0 (4.4)	1.36 (3)	40	(28)
Plumbing (Total Module Incl. Fittings)	2.27 (5.0)	1.73 (3.8)		
Structure	13.62 (30.0)	6.81 (15)		
Total Dry Weight Processing Module	113.18 (249.3)	64.97 (143.1)	410	(269)
Slurry/Effluent	2.27 (5.0)	1.82 (4)		
Total Wet Weight Processing Module	115.45 (254.3)	66.78 (147.1)	410	(269)
Total Processing Module - Volume	0.61 m <sup>3</sup> (17.33 ft <sup>3</sup> )	0.4 m <sup>3</sup> (11.2 ft <sup>3</sup> )		
Total System Weight and Power	202.98 (447.1)	93.34 (205.6)	520	(285)
Total System Volume	0.92m <sup>3</sup> (26.0ft <sup>3</sup> )	0.61m <sup>3</sup> (17.3ft <sup>3</sup> )		

## CORROSION TEST PROGRAM

Materials of construction for the high temperature portion of the wet oxidation system were recognized to be a problem early in the development efforts under contract NAS 1-9183. Corrosion tests were run at that time in a one liter, batch type, stirred reactor with various metal specimens hung on a support structure in the reaction chamber, which was half full of fecal/urine slurry. Hastalloy C-276 was selected as the best material of those tested because there was no evidence of corrosion in six weeks of exposure. As a result of these tests, it was concluded that Hastalloy C-276 would be used for all high temperature portions of the demonstration system to be built under contract NAS 1-11748.

In the preliminary design phase of contract NAS 1-11748, a problem dealing with materials selection became apparent. The corrosion tests conducted under NAS 1-9183 had been run concurrent with development efforts to achieve ammonia removal in the wet oxidation effluent water. Various ammonia removal methods had been investigated including electrolysis, catalytic oxidation, reduction, electrodialysis, and catalyst in wet oxidation. As a result of these studies, the addition of Ruthenium Trichloride to the input slurry was selected as the easiest, most effective means of eliminating the ammonia. Tests showed no measureable amounts of ammonia in the effluent water and a shift in pH from 8.5 to 4.5. This shift from basic to acidic reaction conditions made it necessary to reconsider the materials selection.

Corrosion tests run for another program had shown commercially pure Titanium to be a good metal for acidic wet oxidation reactions. NASA regulations, however, prohibited the use of Titanium in a system utilizing pure oxygen at the required pressure, so a series of corrosion tests were planned using the latest wet oxidation waste model and the ammonia removal catalyst.

### Materials & Specimen Preparation

The nine metals listed below were selected for test based on the previous NASA and other wet oxidation program corrosion studies.

Hastelloy C-276  
E-Brite 26  
MP35N  
Inconel 625  
Zirconium  
Zircaloy 2  
Elgiloy  
Tantalum  
Tantalum - 40% Columbium

Test specimens were fabricated from the nine metal samples as shown by Figure 33 and attached to the one liter stirred reactor head as shown by Figure 34. Ceramic washers were used at the sample support ring and at the "U" bend to electrically isolate the samples from each other and from the reactor body, thereby preventing electrolytic corrosion. The "U" bends were provided to allow stress corrosion testing. The "U" bend legs were deflected to produce a calculated 80 percent of yield stress in the center of the "U". Five specimens were suspended from the support ring at one time.

### Test System & Methods

Figure 35 presents a schematic of the corrosion test setup. An Autoclave Engineers, one liter, stirred, batch reactor consisting of reactor body, heating jacket, reactor head, stirring shaft, magnetic drive, and air motor drive was used to expose the metal specimens to the wet oxidation environment. A shop air supply and regulator were used to drive the air motor at 500 RPM, as measured by a magnetic pickup tachometer. A temperature controller and relays provided normal temperature control to maintain the reactor fluids at 560K (550°F) and to provide automatic shutdown in case of a thermocouple break or over temperature condition. A charging valve and line allowed charging of the reactor with a 5.52 kN/m<sup>2</sup> (800 psig) oxygen precharge. A pressure switch provided system shutdown in the event of an overpressure condition.



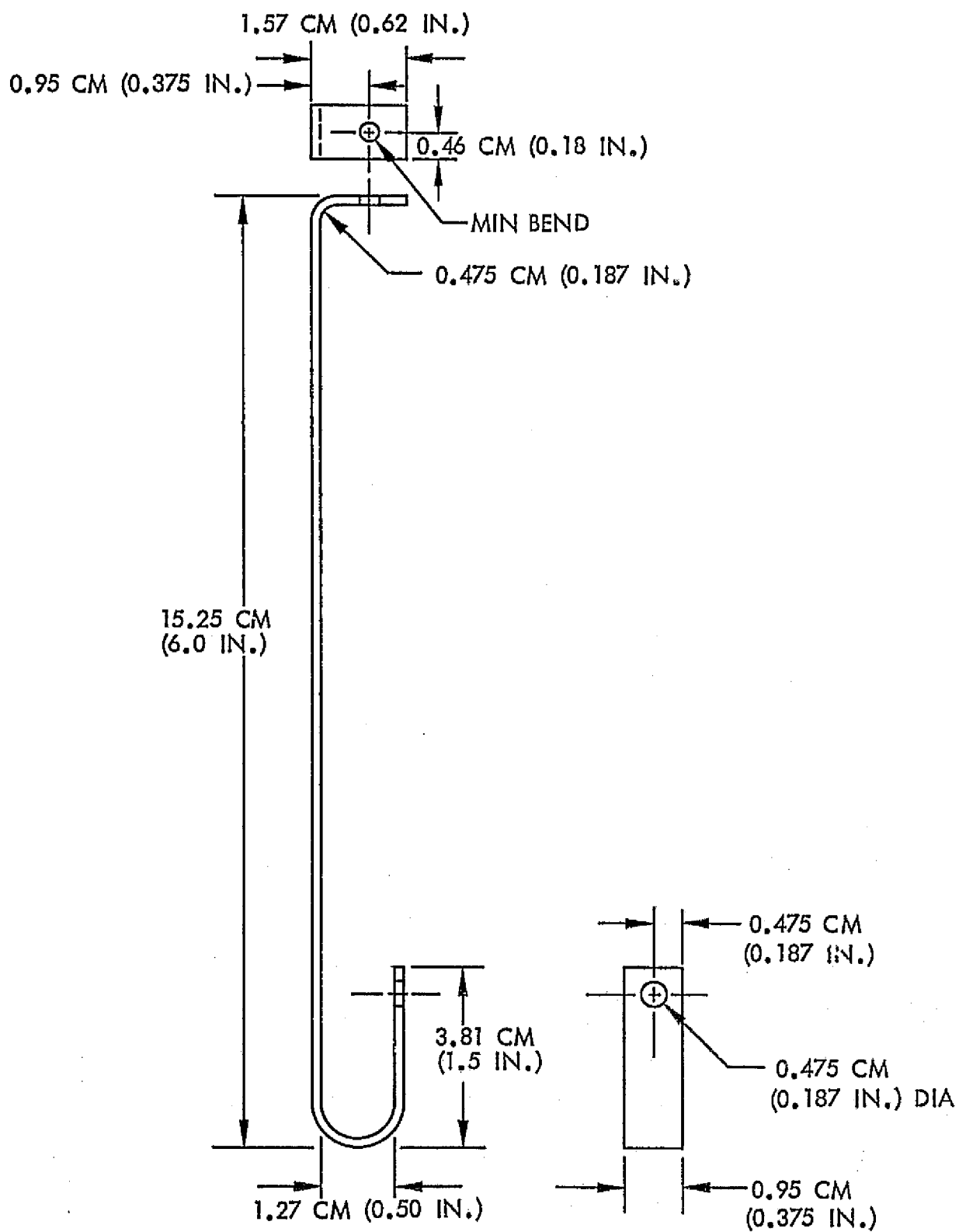


Fig. 33 Corrosion Test Specimen

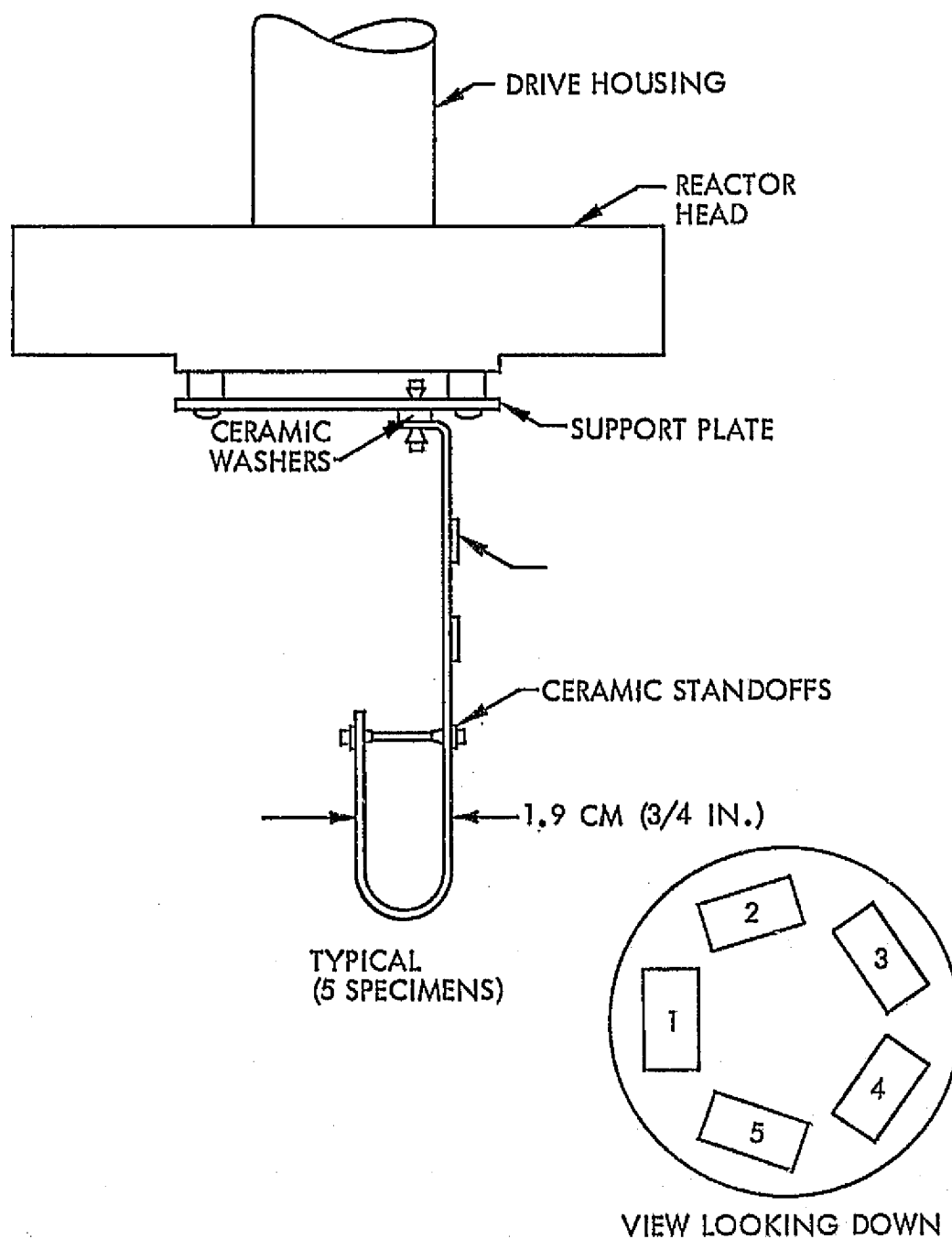


Fig. 34 Corrosion Test Specimen Installation

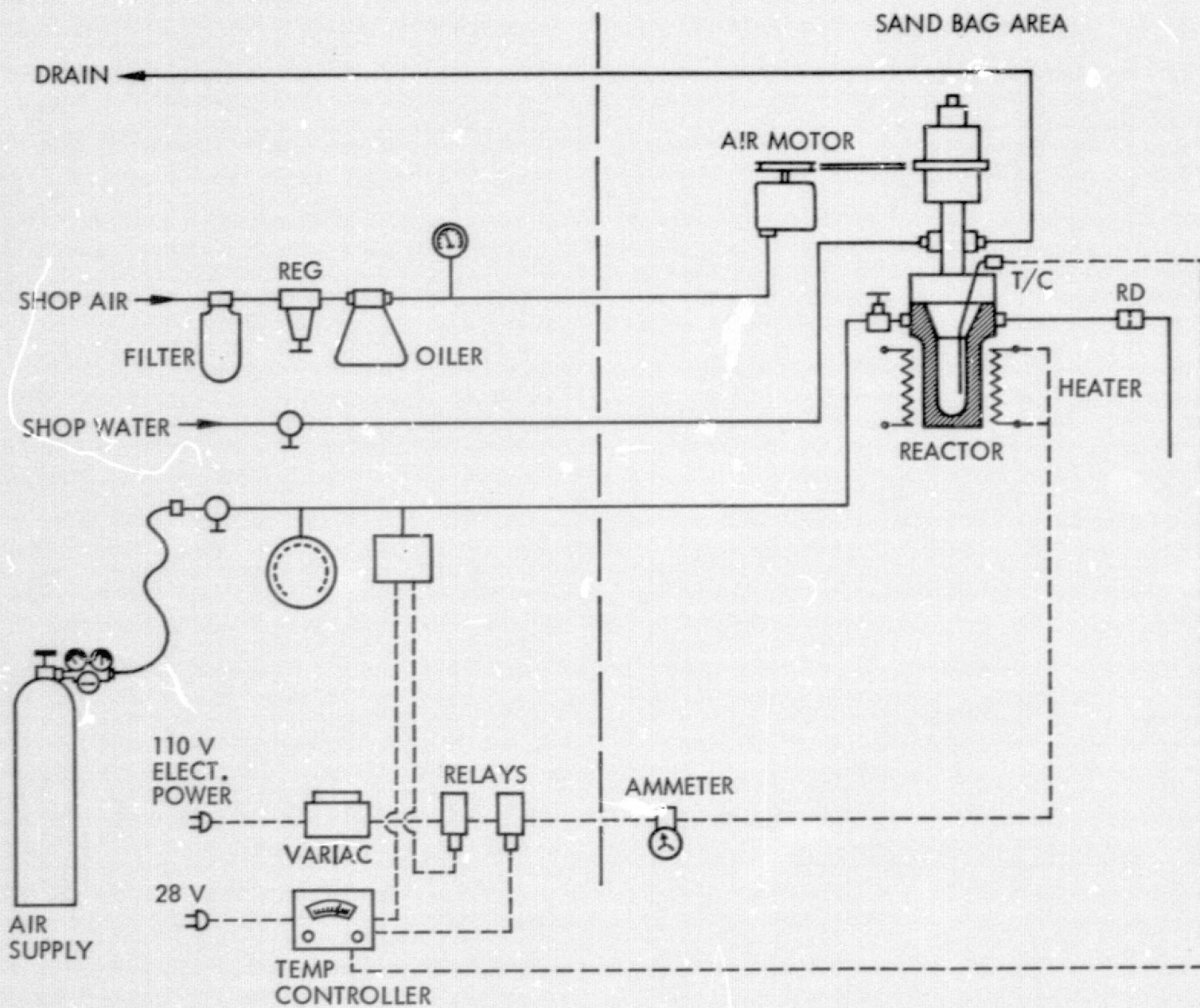


Fig. 35 Corrosion Test Setup

Three tests were run. Two screening tests of four day duration were used to select five metals for a five week test. The tests were run by mounting five test specimens to the reactor head via the support ring, charging the reactor with 460 cc of undiluted fecal/urine slurry, adding 0.1 gm of  $\text{RuCl}_3$ , installing the reactor head, charging with  $5.52 \text{ MN/m}^2$  (800 psig) oxygen, heating the reactor to  $560^\circ\text{K}$  ( $550^\circ\text{F}$ ), and adjusting the drive motor speed to 500 RPM.

#### Test Results

The screening tests produced three metals that showed no evidence of corrosion, i.e., Hastelloy C-276, Tantalum, and the Tantalum/Columbium alloy (WC-640). The results for the remaining six specimens are as follows:

Zirconium	-	Chipped and crumbled
Zircaloy 2	-	Badly corroded in "U" bend area
Elgiloy	}	Generally pitted and cracked
E-Brite 26		
MP35N	}	Two relatively large pits in gas space area of specimens
Inconel 625		

The five week corrosion test was run from August 1, 1973 through September 10, 1973 with three interruptions. The reactor was cooled and opened in order to inspect the specimens on August 20. The specimens were found to be in excellent condition, so the test was restarted on the morning of August 22. On August 27 a pressure switch fitting leak was repaired and on August 28 the rupture disc blew. The rupture disc was only 0.05 mm (0.002") thick and was made of 316 stainless steel, so a slight amount of corrosion weakened it and it blew. A remotely located rupture disc was plumbed into the test setup and the test was resumed on August 30. Inspection of the specimens at the conclusion of the test showed no evidence of corrosion on any of the specimens. Since Tantalum and Columbium are very expensive, difficult to machine, and of very low strength, Hastelloy C-276 was selected for use in the demonstration test system.

## TEST PLANS & PROCEDURES

A test plan and set of test procedures were prepared for the checkout tests and the 45 day demonstration test. The contents of the plan and procedures are summarized in this section of the report.

### Pre-Test Adjustments & Checkout

In preparation for the 45 day test, a number of pre-test settings, calibrations; and checkout tests were planned. The major planned events are described below:

- o Electrical System Checkout - Power off electrical continuity tests followed by power-on operation of each component and subsystem.
- o System Leak Check - Water and Nitrogen gas leak check of entire system at  $3.45 \text{ MN/m}^2$  (500 psig) and at  $10.35 \text{ MN/m}^2$  (2500 psig), except for low pressure elements.
- o Reactor Runup Test - Speed Check of reactor drive system to check smoothness of operation.
- o Oxygen Orifice Calibration and Regulator Adjustment - Measured quantity of oxygen passing through orifice at various upstream pressures with  $15.2 \text{ MN/m}^2$  (2200 psig) downstream pressure.
- o Catalyst Pump Calibration - Run catalyst pump at various stroke settings and measure flow volume.
- o Slurry Pumping System Calibration & Checkout - Fill and bleed bladdered tanks and pumping lines, adjust pressure switch (56), adjust hydraulic pump relief valve (21) setting, hydraulic pump flow calibration, manual cycling of bladdered tank filling and emptying measuring time for each operation to provide data for timer adjustment.
- o Slurry Pump Timer Adjustment - Adjust cams on timer to achieve tank refill in desired sequence and check automatic operation of slurry/pumping system.
- o Sensor and Regulator Adjustments - Adjust settings on reactor pressure switch (47) for both high and low pressure cut off, adjust relief valve (62) setting, adjust effluent water regulator (18) setting, adjust filter  $\Delta P$  switch setting for filter change and automatic safety shutdown, and set isolation valve temperature controllers at  $322^\circ\text{K}$  ( $120^\circ\text{F}$ ).

- o Cold Flow Test - Run complete system at full operating pressure using water and gaseous nitrogen at room temperature.
- o Hot Flow Test - Repeat previous test under full operating temperature and pressure.
- o Final Checkout Test - Repeat previous test using fecal/urine/flush water mixture.
- o Catalyst Loading - Mixup a 22 1/2 day supply of catalyst and charge the catalyst supply tank.

#### 45 Day Test Plan

The forty-five day demonstration test was divided into nine-five day segments. Each segment was planned to be started on Monday and completed on Saturday morning. Each of the nine test segments was devoted to a specific study as follows:

<u>Week No.</u>	<u>Objective</u>
1	Baseline Test Under Nominal Conditions
2	Vary oxygen flowrate
3	Vary catalyst flowrate
4	Vary reactor temperature
5	Vary reactor pressure
6	Vary reactor drive speed
7	Vary slurry flowrate
8	Vary waste composition
9	Baseline test under nominal conditions

It was the intent of the test plan, to provide sufficient data to allow curves of water quality versus each parameter to be drawn. Provisions were made to vary all of the parameters during the test preparation period of the program. The only tasks required to maintain the system in operation, assuming no malfunctions of equipment, were the preparation of the waste slurry, replacement of oxygen bottles, and manual operation of the pulverizer.

## SYSTEM CHECKOUT & CALIBRATION

A number of component and subsystem checkout tests and calibrations listed in the test plans were accomplished prior to the 45 day demonstration test.

### Catalyst System Calibration

The catalyst pump was calibrated by filling the catalyst tank with water, energizing the pump and measuring the water output over a measured time interval. The calibration curve generated by this test is presented by Figure 36. The required catalyst flow rate was a function of the time required to fill the bladdered tanks which was determined by test to be approximately 3.5 minutes. The pump setting and catalyst mix were based on the following data:

Daily Waste Load = 27.8 kgm/day

Number of Bladder Tank Refills = 4/day

Volume of Bladder Tank Refill =  $27.83/4 = 6957$  cc/fill

Catalyst to Slurry Ratio = 1 part/4600 parts

Catalyst Required Per Refill = 1.51 gm

Catalyst/Water Ratio = 1.51 gm/4.34 cc

Volume of Catalyst Solution Per Refill = 4.90 cc

Time to Refill Bladder Tank = 3.5 min

Catalyst Pump Flowrate =  $4.90\text{cc}/3.5\text{min} = 1.4$  cc/min

Figure 36 shows a pump setting of 80 to achieve the desired catalyst solution flowrate.

The volume of catalyst solution placed in the catalyst supply tank was based on providing a 22.5 day supply initially.

Catalyst Supply = 4.9 cc/refill (4) refill/day (22.5) day  
= 441 cc or 136 gm  $\text{RuCl}_3$   
& 391 cc Water

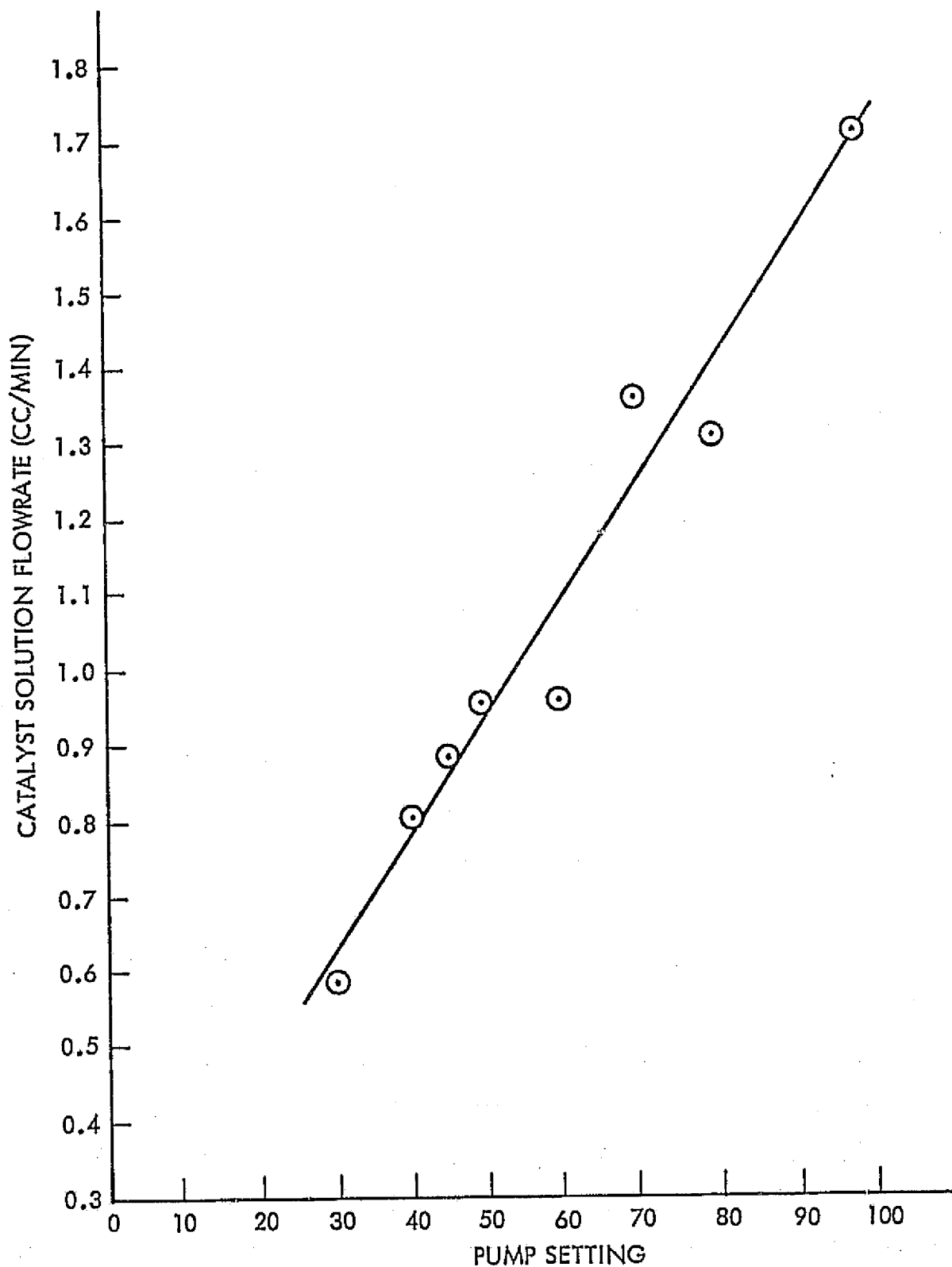


Fig. 36 Catalyst Pump Calibration



A 22.5 day supply was selected so that if something happened to the catalyst an entire 45 day supply would not be lost.

#### Oxygen Flow Calibration

The oxygen supply system checkout test included calibration of the flow control orifice. The calibrations were run by adjusting the orifice upstream pressure to various settings, while maintaining the downstream pressure at  $15.2 \text{ MN/m}^2$  (2200 psi) by adjusting an oxygen bleed valve downstream of the control orifice. A wet test meter was connected to the vent port of the bleed valve. The tests were run with high pressure nitrogen and the results were corrected for oxygen. Three conditions were run; one orifice with downstream pressure at  $15.2 \text{ MN/m}^2$  (2200 psi); and two orifices in series with downstream pressures of  $11.0 \text{ MN/m}^2$  (1600 psig) and  $15.2 \text{ MN/m}^2$  (2200 psig). Figure 37 presents the flow calibration curves generated by these tests. The design oxygen flow rate of 11 pounds/day resulted in an oxygen regulator output pressure of  $16.6 \text{ MN/m}^2$  (2400 psig) based on a nominal reactor pressure of  $11.0 \text{ MN/m}^2$  (2200 psig).

#### Oxygen Pumping System

It was planned to use oxygen from high pressure tanks for testing of the wet oxidation system. A high pressure electrolysis cell was selected during a trade study of oxygen supply sources as the preferred technique for generating oxygen for spacecraft use, but the development of this subsystem was not possible in this contract. Special high pressure  $24.8 \text{ MN/m}^2$  (3600 psig) oxygen bottles were available in the Lockheed Biotechnology Laboratory, but a search for someone to pump them to pressures over  $19.3 \text{ MN/m}^2$  (2800 psi) was not successful. Safety or lack of adequate equipment were given as the reason for not providing this service. A special high pressure pumping system shown by Figure 38 was assembled to pump oxygen from standard laboratory cylinders at a maximum of  $15.2 \text{ MN/m}^2$  (2200 psig) into the special cylinders at  $20.7 \text{ MN/m}^2$  (3000 psig). The system consisted of a single stage piston type oxygen pump, standard oxygen bottle with high pressure regulator, shutoff and bleed valves, pump outlet pyrometer, pressure gage, pressure switch, relief valve, and special  $24.8 \text{ MN/m}^2$  (3600 psi) oxygen bottles. The oxygen pump was capable of pumping the two special oxygen bottles from  $16.6 \text{ MN/m}^2$  (2400 psig) to  $20.7 \text{ MN/m}^2$  (3000 psig) in from 2 to 4 hours depending on the pressure in the standard bottle. The pyrometer, pressure

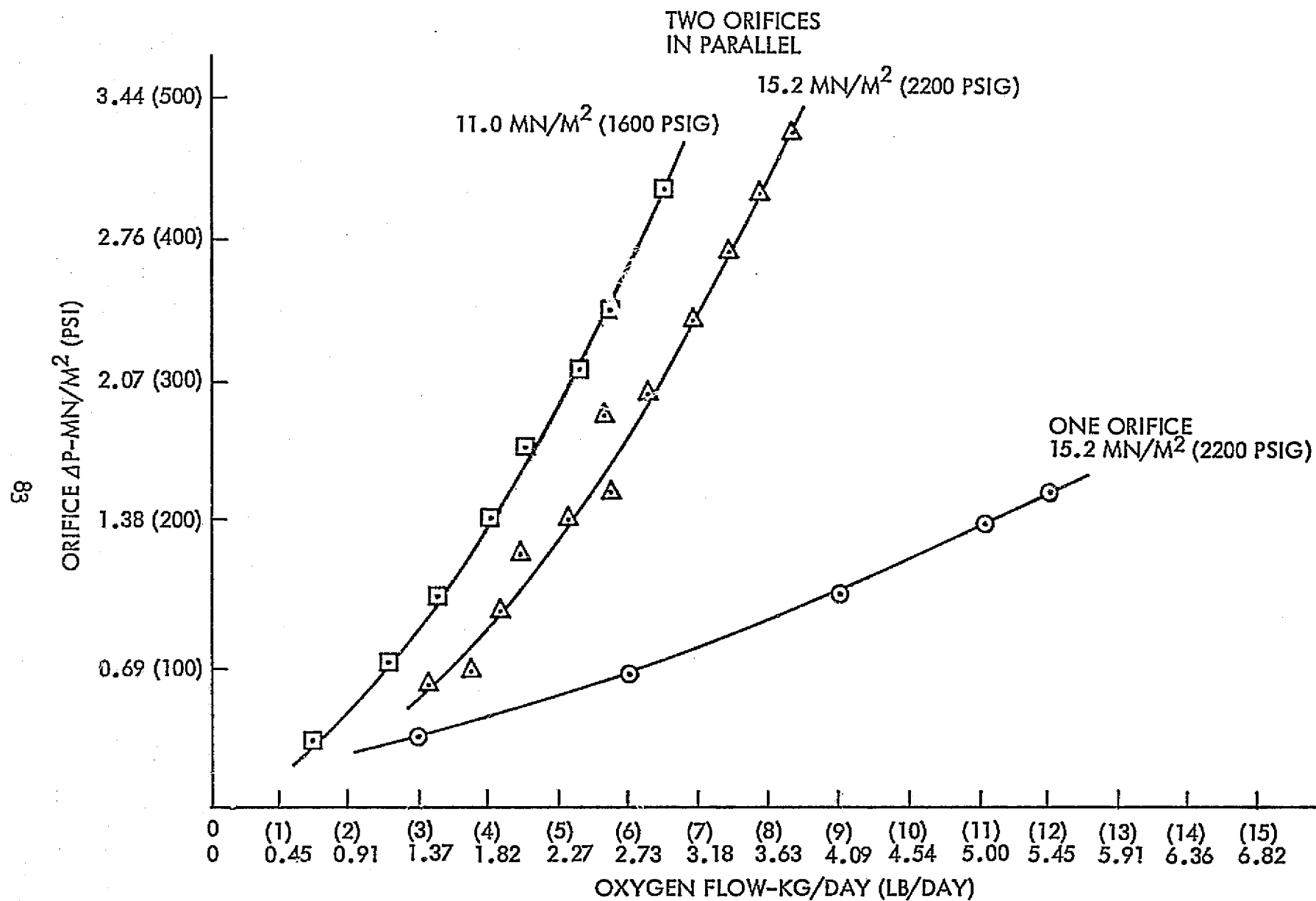


Fig. 37 Oxygen Orifice Calibration

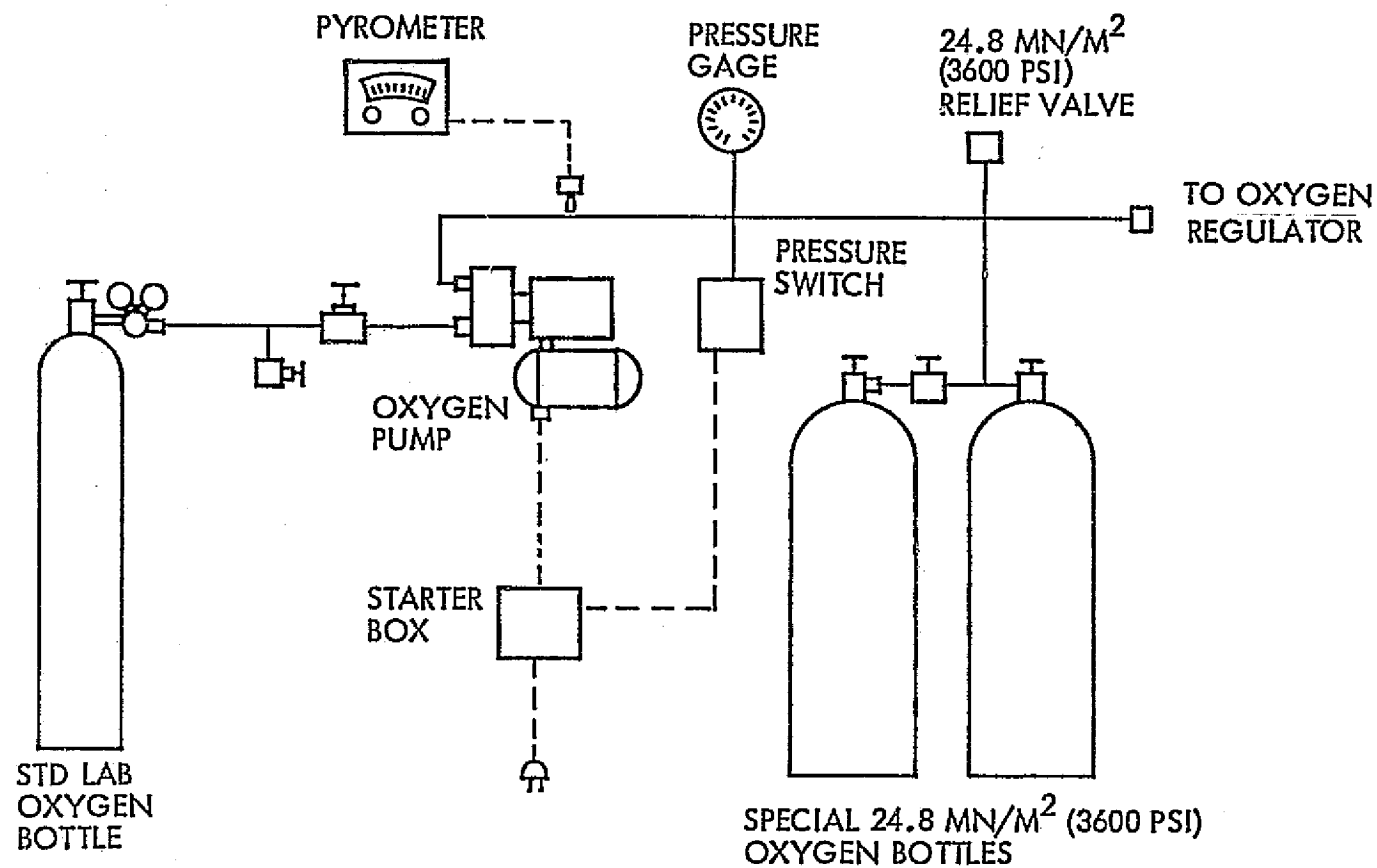


Fig. 38 Oxygen Pumping System

switch, and relief valve were installed as safety provisions to shutdown the pump in case of over pressure and/or temperature and to relieve excess pressure.

#### Slurry Pumping System Calibration & Checkout

The first step in checkout of the slurry pumping system was filling and bleeding of the bladdered tanks and all lines and components. A vacuum pump was connected to the pump bypass valve (20) outlet while a water line and shutoff valve were connected to the pump suction line. After evacuating the system on the back side of the bladdered tanks, the vacuum pump valve was closed and the water supply valve was opened. The slurry side of the bladdered tanks was filled by pouring directly into the tank, since the tanks were mounted with the slurry side up. The pressure switch (56) was adjusted for a low setting of  $690 \text{ MN/m}^2$  (100 psig) and a high setting of  $14.5 \text{ MN/m}^2$  (2100 psig) by operating the hydraulic pump and solenoid valve. The pump relief valve (21) was adjusted to open at  $17.3 \text{ MN/m}^2$  (2500 psig), in the same manner. A pump calibration test was then run by varying the pump setting and measuring the amount of water collected from the pump outlet. A bleed valve attached to the pump outlet was adjusted to maintain the pump outlet pressure at approximately  $15.2 \text{ MN/m}^2$  (2200 psig). Figure 39 presents the results for one pump head. There are four pump heads (two per pump), so that all four could be set at 58 to produce the nominal flow of 1200 cc/min. It was decided to use three pump heads at a setting of 77 each and maintain the remaining head at 0 as a spare.

Following the calibration of the pump, a complete slurry tank filling cycle was executed manually to establish timer sequences. The timer was then adjusted to achieve these time intervals automatically.

Event	Minimum Time Required Between Events	Timer	
		Interval	Elapsed Time
Close Valve No. 11	8 sec to close valve	42 sec	43 sec
Open Pump Bypass Valve	2 sec to bleed tank press.	5 sec	1 min 25 sec
Close $\text{O}_2$ Solenoid	0	21 sec	1 min 30 sec
Open Valve No. 10	0	3 sec	1 min 51 sec
Start Catalyst Pump	3.5 min to fill slurry tank	3 min & 28 sec	1 min 54 sec
Stop Catalyst Pump	0	2 min & 4 sec	5 min 22 sec

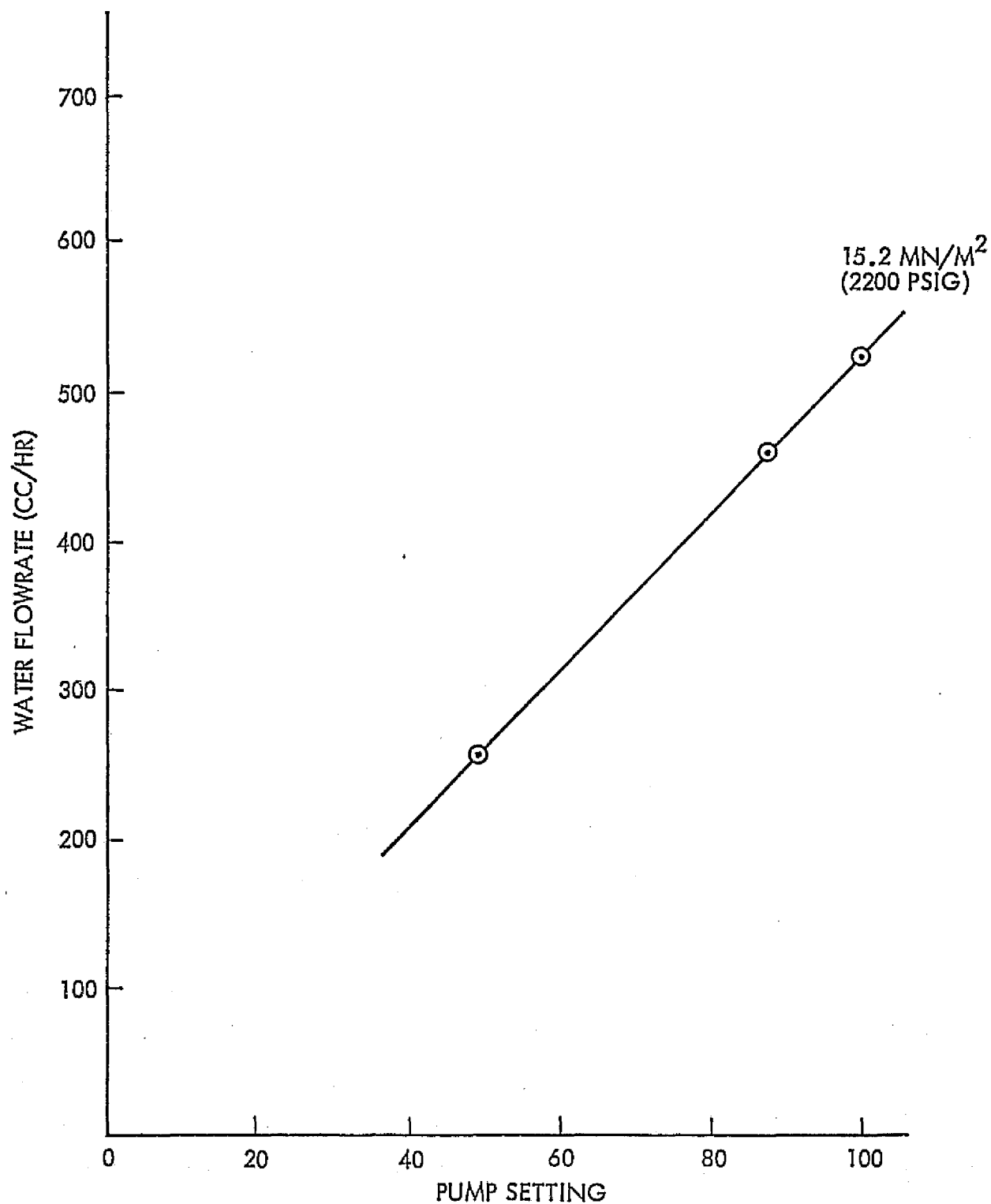


Fig. 39 Hydraulic Pump Calibration

<u>Event</u>		<u>Minimum Time Required Between Events</u>	<u>Timer</u>	
			<u>Interval</u>	<u>Elapsed Time</u>
Close Pump Bypass Valve	}	0	6 sec	7 min 26 sec
Close Valve No. 10		0		7 min 32 sec
Open O <sub>2</sub> Solenoid	}		7 min	
			12 sec.	14 min 44 sec
Open Valve No. 11	}	5 min to pumpup slurry tanks	2 min	
			57 sec	17 min 41 sec

The final timer settings reflected the ability to adjust the cam to the desired setting and the desire to leave ample time for slurry bladdered tank filling and hydraulic pump pressurization of the bladdered tanks prior to opening of valve number 11. During the checkout test, it was determined that the six hour timer did not have sufficient resolution to repeatably and accurately control the slurry pumping system components. The main control timer was changed to a 22 minute timer and a second timer was added to energize the main control timer every six hours. Reducing the main control time to a 22 minute cycle increased the adjustability greatly.

#### Sensor & Regulator Adjustment

Adjustments were made to the controllers and regulators for failsafe shutdown, safety interlocks, and normal operation. These adjustment included:

- o Reactor Pressure Switch High Cutoff 1649 MN/m<sup>2</sup> (2390 psig)
- o Reactor Pressure Switch Low Cutoff 1366 MN/m<sup>2</sup> (1980 psig)
- o Isolation Valve Temperature Controllers 322°K (120°F)
- o Filter ΔP-System Shutdown 4.14 MN/m<sup>2</sup> (60 psi)
- o Filter ΔP-Filter Change 276 MN/m<sup>2</sup> (40 psi)
- o Effluent Water Back Pressure 110 MN/m<sup>2</sup> (16 psig)
- o Slurry Hold Tank Relief Valve 297 MN/m<sup>2</sup> (43 psig) crack,  
235 MN/m<sup>2</sup> (34 psig) reseal

#### Pulverizer Evaluation

Prior to testing the dry waste pulverizer, a specific waste model was defined from the more general waste model listed under system requirements. The 1.14 kgm/day (2.5 lb/day) of food waste, the .59 kgm/day (1.3 lb/day) of wipes, and the 0.36 kgm/day (0.8 lb/day) of housekeeping/hygiene wastes were defined to include:

C 2

Food Wastes	<u>gm/day</u>
Dog Food	362
Aluminized Mylar	91
Polyethylene	408
Polystyrene	182
Paper	91
Wipes	
Cotton Cloth	362
Wash & Dry Pads	227
Housekeeping, Hygiene	
Gauze	35
"Q" Tips	10
Mylar	45
Teflon	45
Disinfectant	90
Paper Towels	136

A series of pulverizer checkout tests were run using the waste materials listed above.

The pulverizer did not accomplish the pulverization which was required. The cotton cloth was particularly difficult to cut and would bind up the pulverizer impellar unless extremely small pieces were fed very slowly. The upper stage cutter clogged very easily because the mylar and cloth would wrap around the center bearing support and stall the pulverizer motor.

A second series of checkout tests, using a 1/3 horsepower motor, and manually precutting the trash, and using only the pulverizer or second stage, improved the pulverizer performance, but still required very slow and careful feeding of the trash to prevent stalling the motor. Three or four hours were required to pulverize the days trash loading. It was concluded at this point in the program, that the trash pulverizer was not capable of providing a supply of ground trash for the 45 day test, so the test must be run with fecal/urine slurry only. It was planned to continue working on the pulverizer so that trash could be introduced into the system near the end of the test.

### Combined System Checkout

The first combined system checkout test was run using water and nitrogen gas under operating pressure, and ambient temperature conditions. Water from the bladdered tanks was pumped through the system by the hydraulic pump system. Nitrogen was introduced through the oxygen system and all elements of the system were operated except for the reactor heaters. During this test all components worked satisfactorily except for the water separator, polishing filter, and reactor drive motor. The water separator vortex sensor was found to be loose, preventing proper signals from being sent to the controller. The reactor drive motor overheated, and a fan blade was added to the motor shaft to provide cooling air flow. No further difficulty was encountered with motor overheating. The pressure drop of the polishing filter was found to be excessive under nominal flow conditions so it was removed. Effluent ash was inspected during testing to determine if the rough filter alone was sufficient, and it was judged to be satisfactory.

On May 14, 1974 a combined system test using fecal/urine slurry and pure oxygen was run. Four effluent water samples were taken with TOC's ranging from 517 to 545. During the test, the oxygen regulator developed an external leak and the replacement temperature controller failed. All other components worked satisfactorily.

The period from May 14 through June 5, 1974 was used to install the oxygen pumping system, replace the temperature controller, repair the oxygen regulator and conduct a hot flow retest using wastes and oxygen. During the retest, the reactor thermocouple failed, so a thermocouple attached to the reactor outlet line was used in all subsequent tests.



## SYSTEM DEMONSTRATION TEST

The system demonstration test was begun following completion of the system checkout tests. The test was started on June 6, 1974 and was completed on September 23, 1974. 737 hours of system operation were completed. In conduct of the test, the system was placed in the automatic mode, which provided totally automatic hands off operation except for manually controlled pumpup of the oxygen bottles and preparation of the input slurry. Once each morning a 24 hour supply of slurry was prepared and loaded into the slurry hold tank and once each afternoon the high pressure oxygen bottles were pumped to a pressure of approximately  $20.7 \text{ MN/m}^2$  (3000 psig.) The following sequence of operations was followed during startup.

- o Place system bypass valve (53) in recycle position
- o Open system isolation valves (8 & 13)
- o Close oxygen and slurry shutoff valves (7 & 12)
- o Energize slurry pumps and bring slurry feed system to reactor pressure
- o Adjust oxygen regulator, if necessary, and energize oxygen solenoid valve
- o Open oxygen shutoff valve (7) and pressurize reactor to approximately 2200 psig
- o Energize reactor heater (high heat mode)
- o Energize reactor drive motor
- o Open slurry shutoff valve (12)
- o After reactor warmup ( $\approx$  30 minutes) position bypass valve (53) in "normal" position
- o Place all panel switches in "AUTO" position

The system was then in automatic mode and unless some failsafe shutdown signal de-energized the complete system, it would operate continuously without adjustment.

Figure 40 summarizes the test by presenting the periods of system operation, operating hours, the cause for system shutdowns, modifications or maintenance performed during the test, the waste loading rate and type used throughout the test, the purpose of each phase of the test and the times that liquid samples were taken.

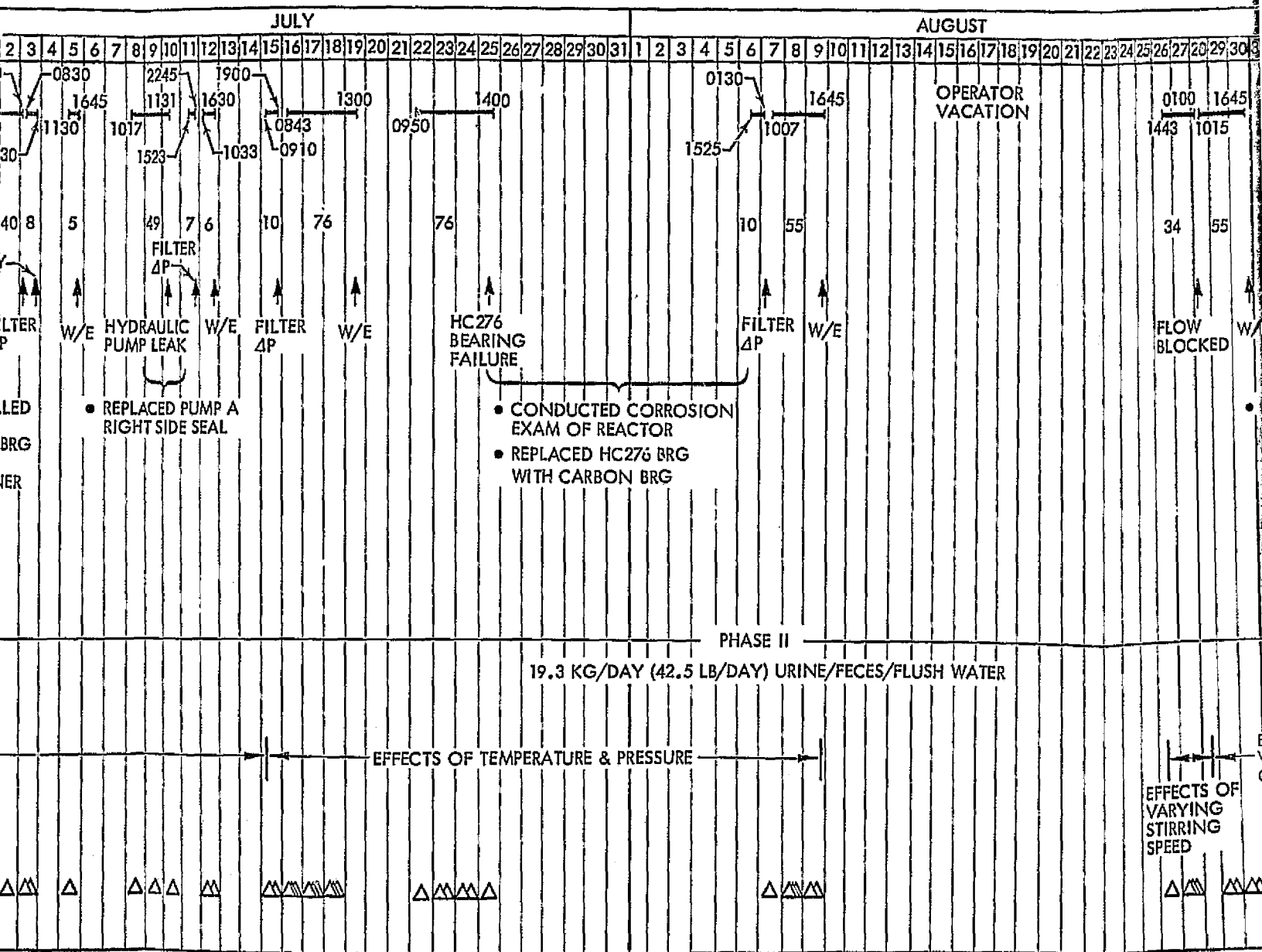
#### Input Wastes

The input waste model was varied throughout the test to reflect different waste quantities, solid concentrations, and waste composition. The compositions of the different waste inputs used in the test program are summarized by Table 8. The two waste models presented in the first two columns were evolved to be used in the test program to achieve the desired variation in waste model parameters. The first column presents a waste mix with high water content and high urine and fecal content. The second column provides a waste mix with higher solids content in as much as food wastes are almost three times higher and are assumed to be made up of aluminized mylar, aluminum foil, polyethylene and semi-solid foods. Total water quantity has been reduced also to provide a thicker mix. The last four columns of Table 8 present four different waste compositions and quantities actually used throughout the test to assess the effects of higher and lower solids concentrations, higher and lower total waste flow rates and because of some equipment problems.

Phase I waste loading - no dry waste - resulted from the failure of the waste pulverizer to provide an adequate supply of pulverized dry waste, so fecal, urine and flush water quantities listed in the first column were increased to provide a full system load by weight. However, there was some concern about using only a fecal/urine/flush water slurry waste, and also there was a desire to evaluate the effects of lower total flow rates. Consequently, the fecal/urine rates were changed to correspond to those of the high solids content waste model (see Table 8), which is now considered appropriate for a space station. This was the Phase II load, and the bulk of the test program was conducted using this waste load.

Phase III and IV were brief test periods and were primarily to evaluate solids concentration and waste feed rate. It was felt that it was absolutely necessary that a portion of the test evaluate a concentrated dry waste load, and that included for instance such waste materials as indicated above. Phase III added wastes to the Phase II urine/fecal/flush water. Phase IV further raised the solids concentration by using the Phase III dry wastes and reducing the flush water and urine.





FOLDOUT FRAME

2

TEST GOAL 1000 HRS



92

Table 8 Demonstration Test  
Input Waste Loading-kgm/day (lb/day)

Waste Material	Input Form of Waste	Low Solids Content Waste Model	High Solids Content Waste Model	Test Phases*			
				I	II	III	IV
Urine	Slurry	9.9(21.7)	8.7(19.1)	15.1(33.3)	8.7(19.1)	8.7(19.1)	4.9(10.85)
Feces		0.77(1.7)	2.5(1.1)	1.2(2.6)	0.5(1.1)	0.5(1.1)	0.39(0.85)
Flush Water		7.6(16.7)	10.1(22.3)	11.6(25.5)	10.1(22.3)	10.1(22.3)	3.8(8.35)
Food Wastes, Housekeeping	Dry Waste	2.1(4.6)	4.1(9.1)	-	-	1.0(2.3)	1.0(2.3)
Wash Water	Water	7.6(16.7)	3.8(8.4)	-	-	-	3.8(8.35)
TOTAL		27.9(61.4)	27.2(60.0)	29.9(61.4)	19.3(42.5)	20.4(45.0)	13.9(30.7)

\*NOTE: The load for Phase I was the contract design waste load rate for six (6) men, but with fecal, urine slurry substituted for the dry waste and wash water portion, i.e., the entire day's load consisted of fecal, urine slurry and equaled 27.9 kgm/day (61.4 lb/day).

The load for Phase II, also, consisted only of fecal, urine slurry but in the proportion specified in a waste load revised recently for space station application. The load was greater per man by about 50%, and so a four (4) man load was about equal to the system design load. The fecal, urine slurry portion was 19.3 kgm/day (42.5 lb/day).

The load for Phase III was the same as Phase II but with the dry waste (not wash water) also included.

The load for Phase IV was one half of the contract design waste load and in the prescribed proportions for all three types of waste - fecal, urine slurry; dry waste; and water. Thus the total load was 13.9 kgm/day (30.7 lb/day).

### Test Summary

The purpose of each period of the test is shown by Figure 40. The initial period from June 6 through July 14 was used to work out system problems and established a baseline under nominal operating conditions. Subsequent periods evaluated the effects of varying system parameters as follows:

<u>Period of Test</u>	<u>Parameters Tested</u>
July 15 through August 9	Pressure and Temperature
August 26 through August 28	Stirring Speed
August 28 through September 5	Oxygen Flow
September 5 through September 17	Slurry Flow
September 17 through September 23	Dry waste included in load

On the second day of the test, the reactor drive tachometer showed zero RPM, indicating that the internal shaft was not rotating. Disassembly of the reactor, following shutdown, showed that the ball bearings had failed. One of the balls had dropped out of the race of the inlet bearing and 10 of the 11 balls had dropped out of the outlet bearing. Surface wear, perhaps aggravated by catalyst attack of the surface, had reduced the size of the balls so that they could fall out of the loading slot in the race. The inlet ball bearing was omitted during reassembly allowing the drive tube carbon bearings to support the inlet end of the stirring shaft. A metal retainer, shown by Figure 41, that slid on the stirring shaft before the bearing was pressed on, was used to prevent the balls from dropping out of the loading slot even though slightly worn. A new outlet bearing was installed and the test was restarted. During pressurization of the reactor with oxygen, a loud crack similar to a burst disc rupture was heard. The reactor drive tachometer was showing zero RPM following the noise, so the system was shutdown and the reactor was disassembled. Inspection of the reactor internals and discussions with the assembly technician resulted in the following conclusions:

- o Some difficulty had been encountered while installing the clam shell baffle assemblies into the reactor, so the technician used several drops of oil on each baffle skid to assist in sliding the clam shells into the reactor.

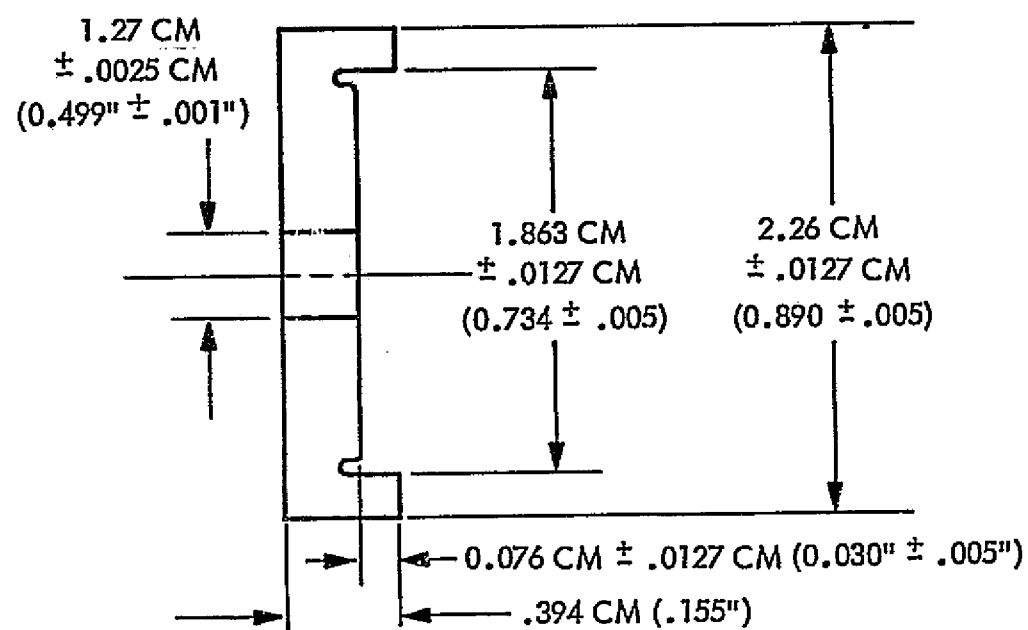


Fig. 41 Ball Bearing Retainer



- o The reactor was usually at least partially full of water or slurry during pressurization, but this time no water was added to the reactor.
- o The pressurization of the empty reactor with pure oxygen resulted in rapid oxidation of the oil behind the baffle assemblies pushing them inward.

Damage to the reactor included overloading of the ball bearing, bending of some of the stirring rods, and buckling of the clam shells and dams. One of the clam shells was repaired and installed along with a new bearing and retainer ring. The bent stirring rods were straightened. However, it was not feasible to repair the clam shells, and the stirring rod would not clear both clam shells when mounted together. So only the bottom shell was used under the assumption that in a one "g" environment it would contribute most to plug flow through the reactor. Performance of the system was checked in subsequent tests to verify this assumption.

On June 20 a flow blockage occurred as evidenced by a rise in slurry pump outlet pressure to the relief valve setting of  $17.3 \text{ MN/m}^3$  (2500 psig). The system was shutdown and flushed with water. No difficulty was encountered in passing water through the system, so the source of the blockage was not discovered. The system was restarted and after one day of testing the flow blockage occurred again. Disassembly of the reactor showed that the inlet bearing spider had rotated approximately 20 degrees, thereby obstructing the inlet line. Grooves were machined in both spider assemblies to allow slurry to flow around the spider should it rotate again.

Inspection of the reactor also showed a generous coating of Ruthenium black on all internal surfaces of the reactor, particularly in the inlet half. Investigation of the characteristics of  $\text{RuCl}_3$  indicated that it probably was breaking down to HCl and Ru black which was displacing metal ions from the surface of the reactor parts. This action evidently made the reactor surfaces catalytic, and it was surmised that only occasional repickling of the reactor need be done to maintain catalytic action.

It was judged by all involved in this test program that testing should be resumed with the system as configured, but that the change in the reactor internal configuration, the problem of pulverizing the dry waste, and the catalyst deposition in the reactor required revision of the test plan. A baseline test was felt to be desirable to determine with the catalyst flow stopped whether water purity would degrade and if so how fast. A reduction in the total load was felt to be in order not only because of the difficulty of providing pulverized dry waste but because of the faulty "plug flow" configuration with only one baffle in the reactor. Consequently, a new test matrix parameter was introduced: waste load-total, and type. Initially, the waste load was only slurry (fecal, urine) and this was to be continued, but the total load was reduced to be commensurate with the reactor effectiveness which was judged qualitatively to be  $2/3$  to  $3/4$  of the original plug flow design. Hence, a new baseline test was started in which catalyst flow was zero, and the slurry load was reduced to 19.3 kgm/day (42.5 lb/day) which is equivalent to the slurry portion of the new load being developed for future space station design purposes. This new quantity is the slurry portion of a four (4) man size waste load rather than six (6) man size and is significantly higher in flush water used per individual. Since a change was planned in the design waste load for future system development, the new load quantity, also, served to bridge between the contract load model and the new load model that would subsequently be used. After one day of testing, another ball bearing failed. The bearings were replaced and the test was restarted. During the night operation of July 2-3, an erroneous signal from the filter  $\Delta P$  switch shutdown the system. Restarting on the morning of the 3rd of July resulted in satisfactory performance.

On July 10, a leak in the piston shaft seal on the right head of hydraulic pump A resulted in a system shutdown. The seal was replaced and the test resumed. Another erroneous filter  $\Delta P$  signal shutdown the system on the night of July 11 and again on July 15.

Two 76 hour periods of uninterrupted testing was achieved between July 16 and July 25 during which time the effects of varying process pressure and temperature were evaluated. On July 25 another bearing failure shut down the system. It was decided at this time to change to carbon bearings because the drive tube carbon bearings were still in excellent condition. The outlet end cap was machined to accept a carbon bushing identical to the ones used in the drive tube. The reactor body was also examined for corrosion by x-ray, dye penetrant and microscopic techniques. The body was found to be sound with only surface effects of the catalyst. Testing was resumed on August 6. Another erroneous filter  $\Delta P$  signal caused shutdown on the night of August 6-7, and the system was restarted satisfactorily the morning of August 7. Testing was suspended between August 10 and August 25 while the chief operator was on vacation.

A flow blockage that initially cleared on restarting the system subsequently caused a shutdown during overnight operation on August 27-28. After weekend shutdown on August 31 and September 1, the hydraulic pump seals were replaced due to leakage observed the previous week. The tight packaging of the pumps resulted in close pumping connections that placed side loads on the pump heads, thereby reducing pump seal life. The pumps were not replumbed at this time, however.

The outlet carbon bearing failed on September 5 and a replacement bearing was installed. The hot reaction fluids attacked the bearing and reduced it to a paste. It was concluded that carbon bearings must be located remote to the hot reaction chamber, since the drive tube bearings were still in good condition. No other system failures were encountered except flow blockage when pulverized dry waste was introduced into the system on September 17. One of the isolation valves was not opening fully and the reactor inlet hole had never been drilled to the full diameter originally specified. Correction of these problems still did not allow the dry waste to be processed through the system. It was evident that much greater attention must be placed on line routing, fittings and component design to allow the slurry, a much thicker paste

with the introduction of the dry waste, to be processed. The thickest dry waste slurry prepared and introduced into the system on the final day of testing September 23, could not be forced from the slurry hold tank into the bladdered tanks of the slurry pumping system.

Conclusions concerning hardware design that can be drawn from the demonstration test are summarized below:

<u>System Shutdown Cause</u>	<u>Number of Occurrences</u>	<u>Design Problem</u>
Reactor Bearing Failure (Ball Bearings)	3	Hastelloy C-276 does not provide adequate hardness to make satisfactory bearing
Reactor Bearing Failure (Carbon Bearings)	1	Carbon bearings for stirrer must be isolated from reactor fluids and temp. as are the drive bearings.
Flow Blockage	5	Reactor spider assemblies rotated blocking inlet and outlet ports. Isolation ball valve did not open fully. Reactor inlet fitting too small.
Filter High $\Delta P$ Signal	4	Incompatible material caused switch corrosion.
Hydraulic Pump Leakage	2	Pump outlet plumbing caused pump head misalignment.

One problem not presented in the test summary discussion, that was encountered early in the test, was intermittent operation of the phase separator. It would operate satisfactorily for several hours and then the gas valve would either stay open or closed. The separator was removed from the system and sent to the supplier for checkout and adjustment. The suppliers' test showed the separator to function satisfactorily. He calibrated the unit and returned it to IMSC. It worked satisfactorily following reinstallation on August 26, 1974.

## Effects of Varying System Parameters on Product Water

Table 9 listed the performance of the system based on effluent Total Organic Carbon (TOC) analysis. Input TOC was measured to be 3505 mg/liter, but was calculated from known COD data to be 10,800 mg/liter. Accurate measurement of TOC is very difficult with high solids concentrations, because such small samples are used (less than 1/2 cc).

Early baseline tests showed an effluent TOC of between 384 and 495, which later dropped to between 213 and 229, probably because it took time for the catalyst to become effective. TOC's between 200 and 300 were characteristic of baseline runs except for two runs conducted during the tests where pressure and temperature were varied. Figure 42 presents a plot of effluent TOC as a function of system pressure for temperatures of 500 and 550°F. Four curves have been drawn because the data was so scattered. Upper and lower extremes of all data points and an average of 500 and 550°F data points with some allowances for baseline data from other tests are shown. Although the data results in curves that are representative of wet oxidation, it is felt that more time is required to reach steady state conditions in the system. Several times during the test program TOC would not increase substantially when temperatures were dropped from 550 to 500°F only to rise sharply the next day under nearly identical conditions, as evidenced by runs on July 23 and 24. The converse was true for August 7 and 8 runs. The data presented by Figure 42 indicate that reduction in process temperature and pressure to 500°F and 2000 psig would result in only slight increases in effluent TOC. These data should be confirmed by additionally testing prior to final selection of process temperature and pressure.

The results obtained from the stirring drive speed tests were most surprising. Little, if any, difference was noted in outlet TOC for any of the speed runs of 0, 300, 400 and 500 RPM. This would indicate that stirring the reactor fluids is not important. This is contrary to results obtained during the previous contract, and theory which indicates agitation should improve mass transfer and particle oxidation. Probably the optimum stirring speed is

Table 9 Effects of Varying System Parameters - Effluent Water TOC

<u>Sample Taken</u>		<u>Process Conditions</u>	<u>Effluent</u>	<u>pH</u>
<u>Date</u>	<u>Time</u>	<u>Being Tested</u>	<u>(TOC/mg/liter)</u>	
June 19	1235	Baseline	385	-
June 20	1430	"	495	-
July 3	1540	"	254	3.1
July 8	1550	"	290	2.9
July 9	0850	"	213	2.5
July 10	0858	"	229	2.9
July 15	1042	532°K, 15.2 MN/m <sup>2</sup> (500°F, 2200 psig)	215	4.5
July 15	1426	532°K, 13.1 MN/m <sup>2</sup> (500°F, 1900 psig)	274	4.0
July 16	0949	Baseline	236	3.5
July 16	1330	534°K, 11.4 MN/m <sup>2</sup> (510°F, 1650 psig)	249	3.5
July 16	1606	560°K, 11.4 MN/m <sup>2</sup> (550°F, 1650 psig)	282	3.5
July 17	0808	560°K, 11.3 MN/m <sup>2</sup> (550°F, 1640 psig)	361	3.5
July 17	1250	532°K, 9 MN/m <sup>2</sup> (500°F, 1300 psig)	566	4.0
July 17	1551	560°K, 8.3 MN/m <sup>2</sup> (550°F, 1200 psig)	604	4.0
July 18	0807	560°K, 8.5 MN/m <sup>2</sup> (550°F, 1230 psig)	492	3.5
July 18	1319	505°K, 8.4 MN/m <sup>2</sup> (450°F, 1220 psig)	693	3.5
July 18	1630	505°K, 10.8 MN/m <sup>2</sup> (450°F, 1570 psig)	805	3.5
July 22	1610	Baseline	425	5.5
July 23	0815	Baseline	241	4.8
July 23	1511	532°K, 10 MN/m <sup>2</sup> (500°F, 1450 psig)	340	3.8
July 24	0812	532°K, 10.4 MN/m <sup>2</sup> (500°F, 1510 psig)	603	4.5
July 24	1555	560°K, 12.4 MN/m <sup>2</sup> (550°F, 1800 psig)	375	5.7
July 25	1026	532°K, 12.4 MN/m <sup>2</sup> (500°F, 1790 psig)	450	3.8
Aug 7	1413	Baseline	400	2.5
Aug 8	0823	560°K, 12.8 MN/m <sup>2</sup> (550°F, 1850 psig)	282	2.8
Aug 8	1252	532°K, 12.8 MN/m <sup>2</sup> (500°F, 1850 psig)	379	2.4
Aug 8	1623	532°K, 9.3 MN/m <sup>2</sup> (500°F, 1330 psig)	675	2.8
Aug 9	0806	532°K, 9.5 MN/m <sup>2</sup> (500°F, 1380 psig)	784	4.0
Aug 9	1412	560°K, 9.5 MN/m <sup>2</sup> (550°F, 1380 psig)	630	4.0
Aug 26	1640	300 RPM	775	6.3
Aug 27	0810	300 RPM	335	3.4
Aug 27	1258	500 RPM	308	3.4
Aug 27	1611	0 RPM	321	3.4
Aug 29	0930	400 RPM	306	3.8
Aug 29	1605	Zero O <sub>2</sub> Flow	300	3.5
Aug 30	0825	Baseline	305	3.5
Aug 30	1615	Zero O <sub>2</sub> Flow	444	5.5
Sept 5	1205	Slurry Flow 1575 cc/hr	362	2.8
Sept 5	1710	Slurry Flow 1200 cc/hr	390	3.2
Sept 9	1526	Slurry Flow 425 cc/hr	764	4.0
Sept 13	1615	Slurry Flow 425 cc/hr	368	2.8
Sept 17	1612	Dry Waste	962	4.0
Sept 18	1338	Dry Waste	516	3.5
Sept 23	1414	Dry Waste	754	3.5
Sept 23	1625	Dry Waste	603	3.5

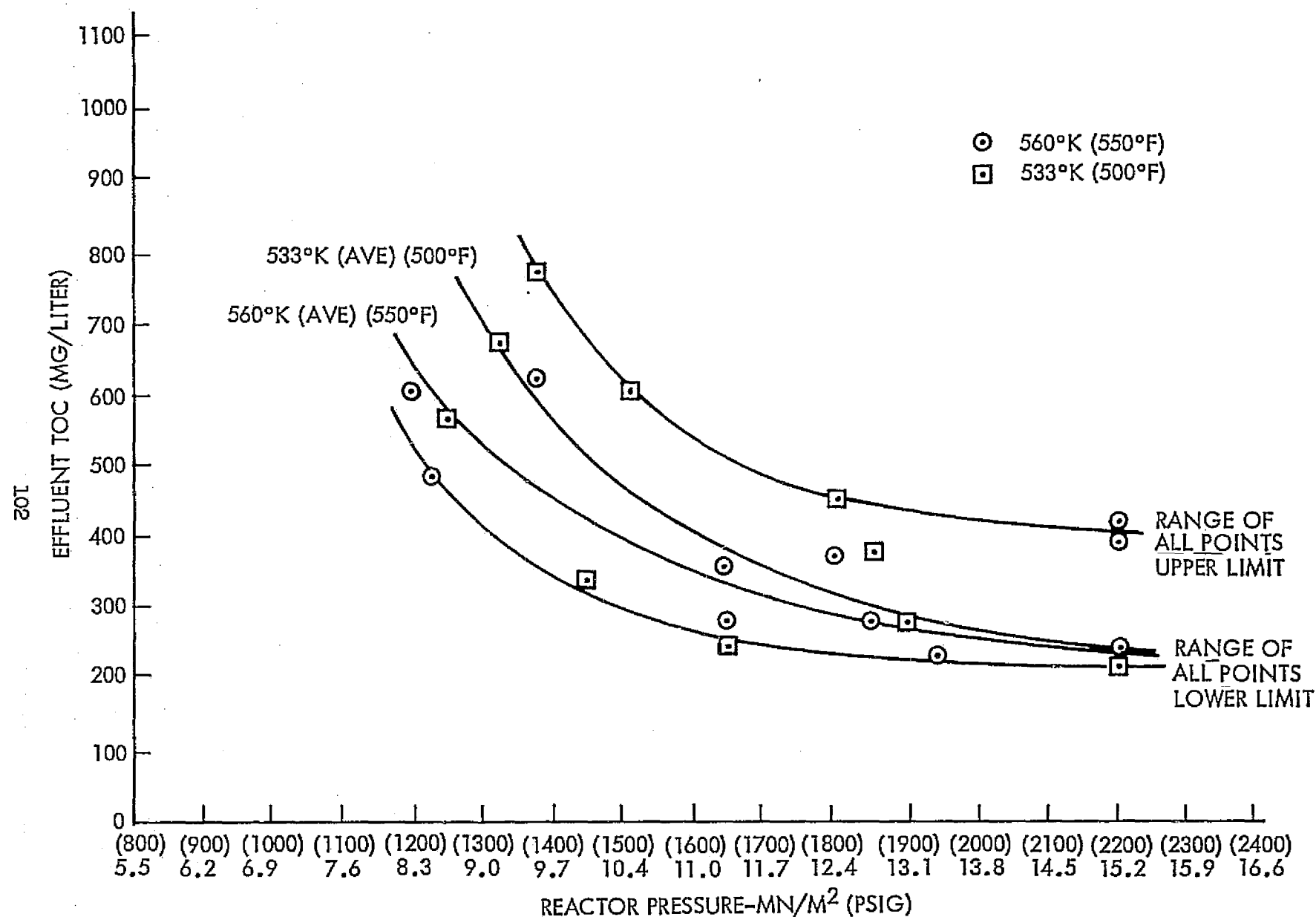


Fig. 42 % Reduction In TOC vs Process Temperature and Pressure

higher than the range tested or equilibrium was not achieved in such a short test period. Similar results were encountered in the oxygen and slurry flow tests where output TOC seemed to be independent of oxygen or slurry flow.

Table 10 lists the gases detected in the effluent gas sample taken on July 18. The only gas contaminants found in the effluent can readily be handled by a spacecraft contaminant control system.



Table 10 Effluent Gas Analysis

July 18, 1974 Sample Time →	Gas Concentration (% by Wt)	
	<u>0832</u>	<u>0910</u>
O <sub>2</sub>	75.4	75.8
N <sub>2</sub>	9.2	7.2
CO <sub>2</sub>	15.5	4.9
NH <sub>3</sub>	≤ 1 ppm	≤ 1 ppm
NO <sub>2</sub>	≤ 0.1 ppm	≤ 0.1 ppm
NO	≤ 0.1 ppm	≤ 0.1 ppm
CO	235 ppm	550 ppm
SO <sub>2</sub>	≤ 1 ppm	≤ 1 ppm
Total HC	89 ppm	42 ppm

## CONCLUSIONS AND RECOMMENDATIONS

The conclusions that have been drawn from the development and testing of the demonstration system deals with process conditions and hardware performance.

- o The system demonstrated the ability to provide a high degree of oxidation by producing a clear, pale yellow liquid effluent with a TOC value between 200 and 300 mg/liter. The color resulted from the  $\text{RuCl}_3$  catalyst addition. The water quality was such that passing it through a vapor compression distillation unit and charcoal should provide potable water.
- o Stirring speed test results from this contract do not confirm previous results which indicated that the use of clam shell baffle assemblies and optimum stirring speed should improve water quality from the 200 to 300 mg/liter TOC value. However, this test was not able to use both baffles.
- o A major design and development effort is required to evolve a satisfactory dry waste pulverizer system capable of producing a pumpable slurry for spacecraft application.
- o The attention given to prevent clogging of the system components and plumbing was not adequate for the thicker trash slurries prepared during this contract effort.
- o The catalyst introduction can be limited to initial pickling of the reactor and perhaps periodic repickling.
- o The majority of the system components including the controls worked reasonably well through the test.
- o The reactor bearings must be isolated from the hot reaction fluids to provide long life.

Based on these conclusions, it is recommended that development of the spacecraft wet oxidation system be continued, and concentrated initially in improving the reactor and trash pulverizer designs. Further steps should be taken to eliminate the clogging problems encountered. This as a minimum would involve redesign of the slurry pump, most component ports and replumbing of the slurry transfer system. Also, new baffle assemblies should be fabricated for the reactor and tests run to determine the effects of agitator speed on effluent water quality in a continuous flow process.

#### REFERENCES

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## LIBRARY CARD ABSTRACT

This report describes the initial hardware development of a spacecraft wet oxidation type waste processing system. The system processes fecal/urine/dry waste slurries in an elevated temperature/pressure oxidation process to convert waste materials to useful products such as water and  $\text{CO}_2$  gas and to reduce the solids to a small inert mass. The system designed, fabricated and tested was based on previous NASA contract efforts that defined the range of and in some cases the specific process conditions suitable for obtaining high conversion rates, and developed component and subsystem hardware designs. The primary objectives of the effort described in this report were to test and demonstrate a wet oxidation system designed to produce a product water requiring minimal processing to be made potable, and to determine appropriate priorities for and types of hardware development required for this process. The program objectives were achieved in a 737 hour demonstration test. Additional development requirements were identified primarily in the reactor, pulverizer, and slurry pump designs.